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Evaluation of Crossflow Microfiltration for Removing Nitrocellulose Fines From Wastewater

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The Radford Army Ammunition Plant (RAAP), Radford, VA is the only Army ammunition plant in the United States that processes nitrocellulose (NC), which is a primary ingredient in military weapon propellants. The NC manufacturing process generates fines that are present in wastewater as suspended solids. Increasingly stringent environmental regulations and a desire to assume environmental leadership have motivated the Army to comprehensively evaluate and develop alternatives to separate, treat, and dispose of NC fines. Research by the U.S. Army Construction Engineering Research Laboratories (USACERL) into separation alternatives has considered: crossflow microfiltration (MF), rotary vacuum filtration, coagulation using synthetic polymer, sedimentation, air flotation, new decanter methods, and other improved operations.

This research investigated crossflow MF for treating NC wastewater by analyzing influential factors and operational parameters, identifying the necessity of pretreatment, recommending an appropriate pretreatment method, and assessing the reuse potential of recovered NC fines and filtered wastewater. The data from this research may be used to develop design criteria for a full-scale MF treatment facility at RAAP.

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The Radford Army Ammunition Plant (RAAP), Radford, VA is the only Army ammunition plant in the United States that processes nitrocellulose (NC), which is a primary ingredient in military weapon propellants. The NC manufacturing process generates fines that are present in wastewater as suspended solids. Increasingly stringent environmental regulations and a desire to assume environmental leadership have motivated the Army to comprehensively evaluate and develop alternatives to separate, treat, and dispose of NC fines. Research by the U.S. Army Construction Engineering Research Laboratories (USACERL) into separation alternatives has considered: crossflow microfiltration (MF), rotary vacuum filtration, coagulation using synthetic polymer, sedimentation, air flotation, new decanter methods, and other improved operations.

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Foreword

This study was conducted for Headquarters, U.S. Army Corps of Engineers under Project 4A162720D048, "Industrial Operations Pollution Control Technology"; Work Unit PF-UZ3, "AMC Wastewater Treatment Plant Sludge Management." The technical monitor was James Heffinger, Army Environmental Center, SFIM-AEC-TSD.

The work was performed by the Pollution Prevention Division (EP) of the Environmental Sustainment Laboratory (EL), U.S. Army Construction Engineering Research Laboratories (USACERL). Dr. Jae K. Park is an assistant professor at the University of Wisconsin at Madison. J.G. Heffinger, Jr., W.J. Worrell, Jr., M.G. DeHart and D.A. Musser were associated with Hercules Aerospace, Inc. Dr. Edgar Smith is Acting Chief, CECER-EP, and William Goran is Chief, CECER-EL. The USACERL technical editor was William J. Wolfe, Information Management Office.

LTC David J. Rehbein is Commander and Acting Director of USACERL, and Dr. Michael J. O'Connor is Technical Director.

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1 Introduction

Background

The Radford Army Ammunition Plant (RAAP), Radford, VA is the only Army ammunition plant in the United States that processes nitrocellulose (NC), which is a primary ingredient in military weapon propellants. The NC manufacturing process generates fines that remain in wastewater as suspended solids. NC manufacture at RAAP produces large quantities of wastewater-containing NC fines in the micron and submicron size range. Increasingly stringent environmental regulations and a desire to be a leader in maintaining environmentally clean practices have lead the Army to comprehensively evaluate and develop technological alternatives to effectively and economically separate NC fines. Research by the U.S. Army Construction Engineering Research Laboratories (USACERL) into separation alternatives has considered: crossflow microfiltration (MF), rotary vacuum filtration, coagulation using synthetic polymer, sedimentation, air floatation, new decanter methods, and other improved operations.

An evaluation of various alternatives for recovering, treating, and disposing of NC fines for the United States Army Toxic and Hazardous Material Agency (Arthur D. Little, Inc., 1987) identified crossflow microfiltration (MF) as the most desirable option. This study provided a more thorough investigation into the use of MF for treating NC wastewater.

Objectives

The objectives of this research were to evaluate the performance of crossflow MF for removal of NC fines from wastewater and to generate information needed to develop design criteria for a full-scale crossflow MF facility.

Approach

This research was conducted in the following steps:

1. NC wastewater streams were characterized in terms of quantity and physical properties of the NC fines before selection of experimental units.

2. Bench-scale and pilot-scale experiments were conducted to investigate the influential factors and operational parameters of crossflow MF for treating NC wastewater.

3. The reuse potential of recovered NC fines and filtered wastewater was analyzed.

4. The hazard potential associated with the crossflow MF process treating NC wastewater was assessed.

5. A specific prefiltration option was recommended.

6. Recommendations were made for safety in design and operation of the filtration process.

Scope

Although the intent of the research was to generate an information base to be used in developing design criteria for a full-scale crossflow MF facility, the long-term performance of MF, such as the life of the membranes, was not experimentally determined.

Mode of Technology Transfer

This research will be used as a basis for planned pilot demonstration projects at RAAP that will determine the best separation, treatment, and disposal methods for NC. It is anticipated that the information derived from this study will be used to develop design criteria for a full-scale crossflow MF facility to be constructed at RAAP.

2 NC Wastewater—Overview

The following two preliminary assessments were conducted prior to the microfiltration (MF) studies: (1) Identification of the sources and the quantities of NC wastewater streams generated at RAAP, and (2) Determination of the particle size distribution and other characteristics of the NC wastewater streams.

Description of NC Manufacture

The process used to manufacture NC involves nitration of cotton linters or woodpulp cellulose with a mixture of nitric and sulfuric acid in a multistep continuous operation. The majority of the residual acid after nitration is removed in a counter-current backwash centrifuge. After centrifugation, the NC product is subjected to a number of subsequent steps. The acidic NC is boiled, cut, and beaten to reduce fiber size and release entrapped acid. Then the NC is poached with soda ash, washed and screened, blended, and washed again at a final wringer operation.

Acidic wash waters containing easily settleable fibers are sent to the boiling tub settling pits. Settled fines from the boiling tub pits are periodically collected, subjected to subsequent NC process treatments, and included in the bulk product for certain NC grades. The poacher, blender, and final wringer wash and transfer waters are discharged to the poacher settling pits. These waters are neutral to slightly alkaline and contain a mix of short fibers and colloidal fines. Settled fines from the poacher pits are blended back with the bulk product for certain NC grades. Wastewater from the poacher pits containing NC fines are then centrifuged with the DeLaval centrifuges, combined with boiling tub pit discharge water, and then discharged to lime neutralization and the settling lagoons. The National Pollutant Discharge Elimination System presently sets the limitation for total suspended solids in wastewater at 40 mg/L; the RAAP pollution abatement system currently complies with this standard. It is anticipated that the State of Virginia may impose a more stringent discharge limit in the future.

Water Usage Survey

A water survey was conducted of the NC production process from nitration through the final wringer. Since flow meters are not available on the influent and effluent lines of the processing tubs in each of the process buildings, water usage rates were estimated based on operator interviews and observed water levels in process tubs.

The water usage rates were found to vary over a wide range depending upon the skills of the operators, the weather, and the types and final blends of NC being produced. For example, the number of transfers required to move all of the NC out of a boiling tub depends, in part, on the skill of the operators in using fire hoses to divide the material and flush it through the piping to the beater house without plugging the lines. Also, during freezing weather, water in many of the NC production areas is allowed to flow continuously for freeze protection. Taking this variability into consideration, flow diagrams were developed based on best engineering estimates.

Based on production records, 17.4 tubs/month (0.870 tubs/day) of cotton linters at 23,000 lb/tub* were produced in 1992. In accordance with the water balance for cotton linters, a total of 812,300 gal of water is estimated to be discharged for each tub of NC produced for a total of 14,134,000 gal/month. Also, in 1992, 15.3 tubs/month (0.765 tubs/day) of pulp at 33,000 lb/tub were produced. In accordance with the water balance for pulp, 1,231,900 gal of water is estimated to be discharged for each tub for a total of 18,848,070 gal/month. The combined total divided by 20 working days/month yields an average water discharge rate of 1,649,000 gal/day. Estimated water discharge rates for each purification operation are summarized in Table 1.

Table 1. Estimated water discharge rates for each purification operation.

Location	Linters (gal/day)	Pulp (gal/day)	Subtotal (gal/day)
Boiling tub	209,583	321,989	531,572
Beater	161,385	160,880	322,265
Poacher (drain)	41,647	54,927	96,574
Poacher (decant)	124,958	164,169	289,127
Blender	43,065	56,610	99,675
Wringer	126,063	183,600	309,663
Total			**1,648,976**

* 1 lb = 0.453 kg; 1 gal = 3.78 L.

As a check on the accuracy of the water balances, records of water discharged monthly at C-line outfall 005 and B-line outfall 007 during 1992 were used to calculate a combined daily average of 3,883,000 gal. It is estimated that NC production accounts for 40 percent of the daily discharge. At the rate of 3,883,000 gal/day, 40 percent is equivalent to 1,550,000 gal/day, or 6 percent less than the daily average calculated from the water balance.

The accuracy of the water balance was also checked by comparing the estimated flow through the DeLaval centrifuges with the designed flow rating of the centrifuges. In accordance with the water balance and 1992 production records, an estimated 1,117,380 gal of water per day was projected to flow through the centrifuges. In comparison, four of eight centrifuges are typically on line 24 hours per day, 5 days per week. At a rated capacity of 166 gpm each, a total of 956,200 gal of water was theoretically processed per day in 1992, 14 percent less than the amount predicted from the water balance.

Considering the variability that exists in the water usage rate during production of NC and the inexact methods of estimating water flow rates, an error of ±6 to 14 percent is within reasonable limits.

Particle Size Distribution

The amount of NC discharged from each process location greatly varies and the particle size of the NC also varies. Poacher and wringer operations discharge relatively large amounts of NC. Hercules Aerospace Inc.* was tasked to analyze the physical characteristics of NC fines in wastewater and the results are summarized as follows.

Samples representing current production were collected from six different wastewater process locations: (1) influent and effluent for A-line/B-line boiling tub pit, (2) influent and effluent for C-line poacher settling pit, (3) influent to hill tank (storage), (4) effluent (two locations) for the DeLaval centrifuges, (5) C-line influent to the neutralization basin, and (6) C-line effluent from the settling lagoon.

Five equal samples from each location were collected at different times and mixed to obtain representative composite samples. NC particle size distributions were evaluated by passing sample material through filters of various pore diameters (420, 105, 75, 44, and 0.45 μm) and determining the weight of the collected material.

* Hercules Aerospace, Inc. is located at Radford Army Ammunition Plant, VA.

Particle size distributions of the NC collected on the 44 and 0.45 μm filters were determined using an optical microscope. The data are shown in Table 2. No significant differences were observed in particle size distributions of the NC collected on the two smaller pore diameter filters.

Table 2 also shows the pH and total suspended solids (TSS) data for each of the sampled locations. The table shows that the pH is neutral with the exceptions of the acidic waters from the boiling tub pit and the basic waters of the influent to the hill tanks (part of the water recycle system). The TSS and particle size distribution data displayed in Table 2 show that pH exerts a major influence in the settling of NC fines. The samples from locations with low pH values, such as the boiling tub house samples (pH 1.6), had low concentrations of NC as TSS. Additionally, these extreme pH samples contained relatively low concentrations of the larger NC fines and high concentrations of very small fines. After settling, the extreme pH samples appeared clear while the neutral pH samples were cloudy. Further research efforts will clarify the relationship between pH and settling concentrations of NC fines.

Quantitation of the TSS and particle size distribution of the NC fines at these process locations resulted in the following significant conclusions: (1) the TSS at each of the sampled locations were less than 100 ppm with the exception of the influent to the poacher pits and the poacher pit effluent (centrifuge influent), and (2) the vast majority of the NC fines were small enough to pass through a filter with 44 μm pore size but were retained on the 0.45 μm filter. Interestingly, these experiments were performed on a weight average basis; if the distributions were on a number average basis, the results would be skewed much further to the smaller particle sizes.

Table 2. pH, TSS, and size distribution (%) of NC based on weight of material retained on standard sieves.

			Filter Pore Diameter (μm)				
Sample	**pH**	**TSS mg/L**	**420**	**105**	**75**	**44**	**0.45**
Boil tub influent	1.6	45.2	0.0	29.3	0.0	0.0	70.7
Boil tub effluent	1.6	29.8	1.3	21.8	7.9	4.4	64.6
Poach pits influent	6.9	457.6	9.5	31.1	1.7	3.5	54.2
Hill tank influent	9.5	92.2	1.7	2.3	0.6	1.2	94.2
Centrifuge influent	7.0	148.0	1.0	4.3	2.3	2.9	89.5
Centrifuge effluent	7.1	72.0	1.2	4.3	3.7	4.0	86.8
Pit 3020	7.2	18.6	22.1	8.0	1.8	0.9	67.2
Neutral influent	7.8	24.6	11.7	15.2	2.3	6.4	64.3
Lagoon effluent	7.7	26.4	32.6	4.2	0.0	1.1	62.1

3 Background on Crossflow MF

Crossflow MF vs. Other Separation Technologies

Crossflow microfiltration (MF) is one of the latest membrane filtration processes. Particles that can be removed by crossflow MF are within the range of 0.1 to 10 µm (Gregor, 1988). The performance of crossflow MF compares to other processes as follows:

1. The quality of permeate is more consistent than with conventional granular filtration
2. Less clogging and lower filtration rate drop over time than with dead end MF
3. Lower pressures required than with ultrafiltration (UF and reverse osmosis)
4. Better performance than centrifugation where the difference in densities of particles and liquid is small (Mackay and Salusbury, 1988)
5. Higher capital cost than conventional centrifugation but lower maintenance cost
6. Fluxes higher than 100 $L/M^2/hr$ to be competitive with centrifugation (Defrise and Gekas 1988; Short 1988).

MF Membranes

A large variety of membranes are now available for crossflow MF. These membranes can be classified into two broad categories: organic and inorganic membranes. Organic MF membranes are usually made of modified or unmodified organic polymeric materials. Inorganic materials that are used to make membranes include aluminum, zirconium oxide, carbon and carbides, stainless steel, nickel, etc. (Meares 1987). Surveys of commercially available crossflow MF membranes and crossflow MF systems were made by Defrise and Gekas (1988) and van der Horst and Hanemaaijer (1990). The morphology of membranes was discussed by van der Horst and Hanemaaijer (1990). Asymmetric membranes, which are characterized by a relatively dense and

thin top layer and an "open" support structure, were considered to be more suitable for crossflow operation than symmetric membranes. However, straight-pore symmetric membranes were also considered to be suitable for operation in a crossflow mode.

The most important criteria affecting the selection of a MF membrane for a certain application are:

1. Charge
2. Hydrophilicity/hydrophobicity
3. Mechanical strength
4. Chemical and thermal resistance
5. Sterilizability
6. Support (most MF membranes are available as unsupported films or case on nonwoven PET or polyolefin)
7 Pore size distribution
8. Porosity and tortuosity
9. Reproducibility
10. Cost.

Crossflow MF Modules

Major modules used in crossflow MF include: (1) tabular, (2) hollow fiber, (3) spiral wound, and (4) plate and frame, or flat sheet (Figure 1). A comparison of characteristics of crossflow MF module configurations was given by Mackay and Salusbury (1988), and is summarized in Table 3.

In a tubular module, filtration membranes and their housings are of the tube shape (Figure 1a). Feed enters from one end of the tube and leaves as concentate from the other end. Permeate passes out through the walls of the membrane. Tubular modules can have one or more polymer tubes in an encasing. The tube diameters usually range from 2.5 to 25 mm. Hollow fiber modules consist of small tubes with internal diameters from 0.25 to 1 mm. They function in the same way as tubular modules.

In a plate and frame module, membranes are attached to rigid plate-like supports stacked together in a casing. Plates have grooves to collect the filtrate. The spacing between adjacent plates varies from 0.25 to 2.5 mm. In thicker channels, baffles may be provided to create turbulence. Feed enters from one end in these spaces and leaves

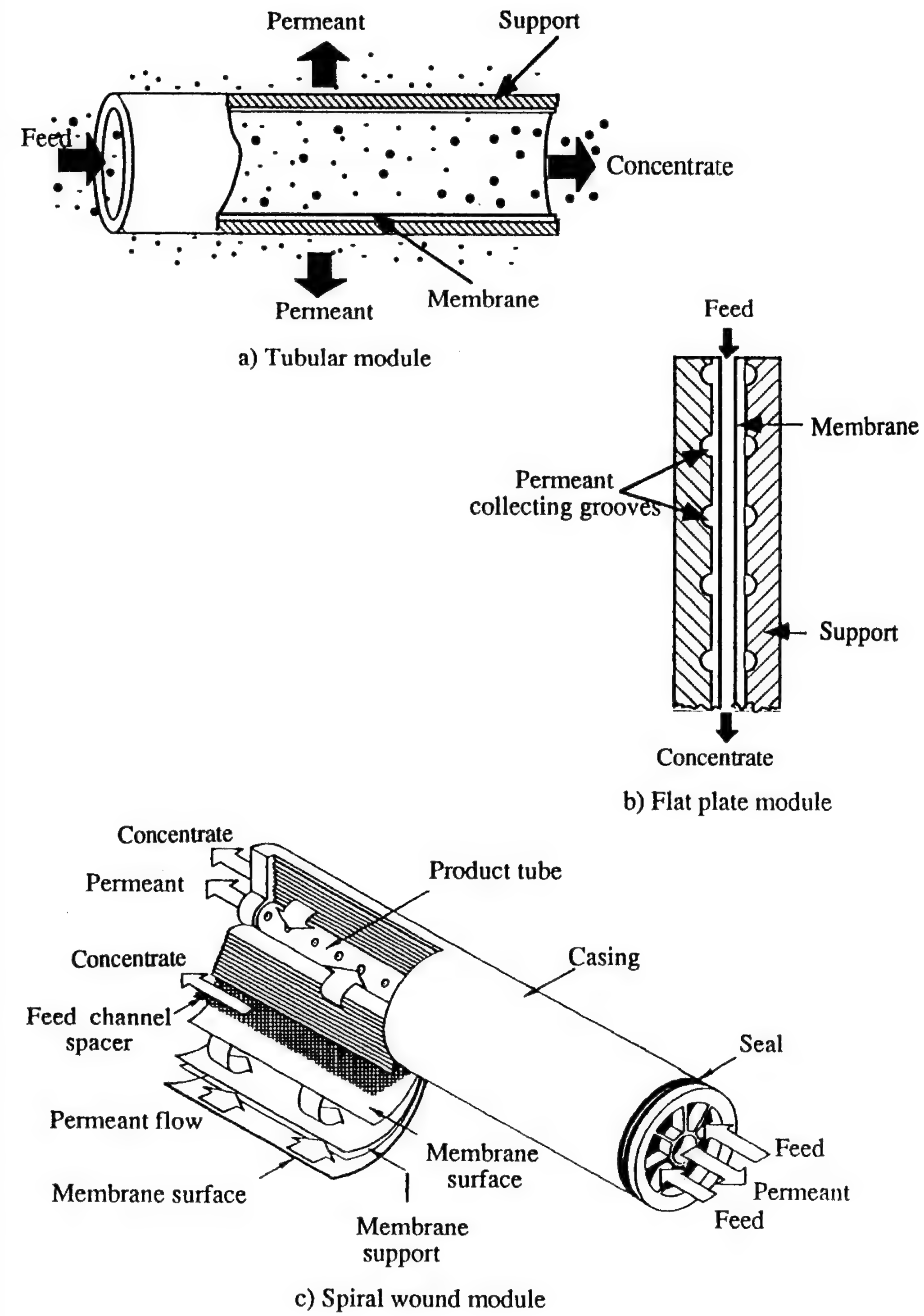

Figure 1. Crossflow microfiltration modules.

as concentrate from the other end. Permeate passes through the membranes into grooves in the plate that carry it to the collection channel (Figure 1b).

In a spiral MF module, the support and a material to carry the filtrate are wound together in a spiral shape and enclosed in a cylindrical casing (Figure 1c). Spacing for passing feed is kept between 0.25 and 0.5 mm. Feed flows through this module along the longitudinal axis of the cylinder. Permeate passes through the membrane into the permeate collection layer, in which it flows spirally towards the collection tube at the center of the cylinder.

Tubular and flat plate modules are, in general, more suitable for wastewater treatment because they have fewer clogging problems. In addition, the membranes are easier to clean and replace. However, energy consumption in these modules is higher than in spiral modules. Tubular modules can be cleaned mechanically as well as chemically.

A modular design with Accurel® microfilter tubes was described by Schneider and Klein (1982). It is a kind of hollow thread, capillary, or tubular membranes in the form of bundles made into separation modules. Usually these modules are operated with transmembrane differential pressure of 1 to 2 bar and a backflush pressure of 0.5 to 1 bar above the former. It was possible to achieve flow rates of super pure water of 1,000 to 10,000 $L/m^2/hr \cdot bar$ depending on the thickness of the membrane.

Hart and Squires (1985) reported that an integrally woven tubular support system with a calcium carbonate precoat on the tubes had been developed to provide a modular, robust, and self-supporting crossflow MF system. Full-scale investigation of such a system was conducted by Treffry-Goatley et al. (1987) using a prototype unit. The unit was operated at a water treatment plant to produce a water of potable standard from highly polluted river water. The cleaning of filters by an external high pressure water spray was effective.

A prototype module model BDF-01 of an axially rotating filter from Sulzer AG (Kroner et al. 1987) improved the dynamic filtration of microbial suspension. Because of its different mode to generate shear at membrane surface, observed flux during crossflow filtration of microbial suspension was significantly higher. Riesmeire et al. (1990) tested the applicability of a mathematical model to predict performance of commercial modules and concluded that construction of MF modules should receive more attention to use the inherent potential of membranes.

Table 3. Characteristics of crossflow microfiltration module configurations.

Tubular	
	1. Large channels - less prone to blockage
	2. Crossflow velocity 2 - 6m/s
	3. Reynolds number > 10^4
	4. Easy to clean
	5. Relatively simple membrane replacement
	6. Low surface area to volume ratio - high space requirement.
	7. High liquid hold-up
	8. High energy consumption
Hollow fibre	
	1. Narrow channels - blockage problem
	2. Crossflow velocity 0.5 - 2.5 m/s
	3. Reynolds number 500 - 3000
	4. Able to be backflushed
	5. High wall shear rates 4000 - 14,000 l/s
	6. Hight surface area to volume ration
	7. Low liquid hold-up
	8. Low energy consumption
	9. Maximum operating pressure limited to approx. 30 psi
	10. High membrane replacement cost
Spiral wound	
	1. Narrow channels prone to blockage
	2. Low capital cost
	3. High surface area to volume ration
	4. Reasonably economical
	5. Design susceptible to collapse
	6. Operates best with clean feed, i.e., little or no suspended matter
Flat place	
	1. Crossflow velocity - 2 m/s
	2. Relatively simple membrane replacement
	3. Energy consumption lies roughly between spiral wound and tubular designs
	4. Visual observation of permeate from each membrane pair is possible

Crossflow MF Operation Systems

Filtration systems are, in general, divided into two types in Figure 2: (1) batch systems and (2) feed and bleed systems. In a batch system, the filtration module is supplied from a constant volume tank. Concentrate passing through the MF unit is recycled back into the system. The level in the tank is kept constant by fresh feed. Recycling is continued until the required recovery is achieved at which time the run is stopped and the storage tank is emptied (Figure 2a). In a batch system, the permeation rate decreases with time due to an increase in the concentration of the recycling stream. This system is usually used for small application or pilot plant studies (Short 1988).

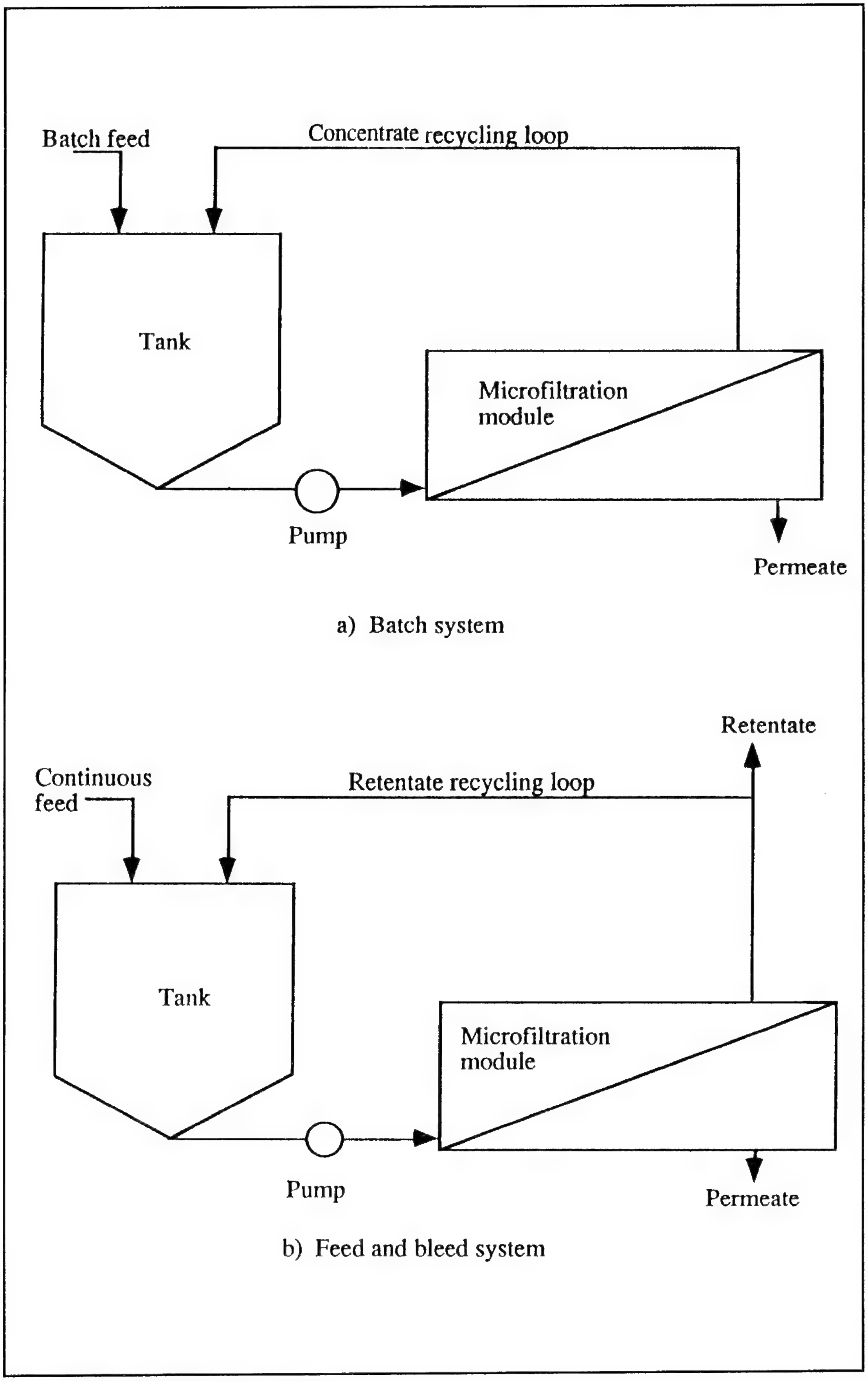

Figure 2. Types of filtration systems.

A feed-and-bleed system operates on a continuous mode. Concentrate is continuously removed from the system. The units of concentrate removal, feed supply, and recycling are such that the concentration in the stream passing through the MF unit is kept at the required recovery value (Figure 2b). A number of these units can be used in series or in parallel to optimize the design.

A modified batch system is the so-called high-resolution tangential flow filtration (HRTF), where a second pump is added to the system. This pump is placed at the filtrate channel so that filtrate rate can be controlled independently of crossflow and fouling can be reduced (Ludwig and O'Shaughnessey 1989). A similar unit was presented by Amicon Division, W.R. Grace Co., which includes circulation and permeate peristaltic pumps as well as LCD display for transmembrane pressure (Technical Data, Amicon. Publication No. 959).

The feed-and-bleed system operates on a continuous mode. In this mode, the feed rate balances the permeate and bleed rates. The concentrate is maintained at the desired concentration. The system is generally designed as stages-in-series to yield better efficiency from a membrane standpoint. However, at some point, the additional cost of the added stages will offset the greater membrane efficiency.

Membrane Fouling and Cleaning

The crossflow leads to distinctly extended filter life, i.e., the flow of a permeate decreases much more slowly as a function of the time. However, it should be noted that crossflow alone will not be able to maintain a sufficiently high permeate flow rate over an extended period of time. Under processing conditions, the flux of crossflow MF membrane may be reduced by an order of magnitude or more from a clean membrane on pure water. Short (1988) concluded that the possible resistance to solvent transport during processing includes: (1) physical blocking of the pore of the membranes, (2) adsorption of material on the membrane surface (fouling) that is irreversible without chemical cleaning, and (3) polarization, or gel layer formation.

Schneider and Klein (1982) pointed out that, in most cases, broad grain size distribution of the suspended particles, where superfine particles can penetrate the membrane pores, caused blocking. A recent experiment by Vigneswaran and Pandey (1988) showed that the percentage of membrane pores blocked depended very much on the suspension particle size and membrane pore size.

van der Horst and Hanemaaijer (1990) discussed the effects of pore size distribution, pore morphology, and process configuration on the separation characteristics and the

membrane fouling. It was concluded that the membrane morphology plays an important role in fouling together with fluid mechanism rather than the adsorption/adhesion of solute onto the membrane surface although this might be true for UF. The smoothness of the membrane surface also determines the effectiveness of crossflow in promoting the transport of retained particles and solute from the membrane surface into the bulk solution, and hence reducing the rate of cake-layer deposition. Furthermore, the internal pore morphology has a large effect on the blockage of pores.

A systematic fouling study was conducted by Gekas and Hallström (1990), who summarized all the possible techniques of membrane cleaning. The study demonstrated the importance of interactions between MF membranes and the substances filtered and also proposed some successful methods of fighting fouling and prolonging membrane life applied in industry. In industrial applications, two methods, backflushing and periodic cleaning, are regularly used. Hydraulic cleaning (rinsing with water) is to be preferred whereas the use of chemicals is recommended only in rare cases.

Optimizing the transmembrane pressure and circulation velocity has been a successful method of preventing fouling (Malmberg and Holm, 1988). High fluxes and good separation were obtained by a process where transmembrane pressure along a membrane channel is carefully controlled.

Experiments on the performance of MF membranes compared with an anisotropic UF membrane during cell harvesting of *E. coli* showed the influence of membrane structure on the composition of deposit layer (Gatenbolm, Fell, and Fane 1988). Transmission electronic microscopy was used to examine the nature of the deposits on the remaining surface. The results showed that, under crossflow conditions, bacteria cells were washed away from the smooth surface of the UF membrane but formed a densely packed layer on the surface of the MF membrane. There was an indication that severe pore blockage might initially occur since many cells were trapped vertically in the pores of the membranes. The cells transported at a greater rate to the membrane surface could be responsible for the build-up of cell mass at MF membrane. In addition, pore size of the membranes affected the blockage of pores by cells.

Defrise and Gekas (1988) reviewed the mechanisms of microbial adhesion and biofouling of crossflow MF in biotechnological downstream processing. Models describing the mechanisms of microbial adhesion to MF membranes were analyzed. It was noted that the microbial adhesion to a MF membrane is a very complex phenomenon, and the effect of the driving force on the microbial adhesion must be

taken into account. Many parameters and mechanisms are important in crossflow MF; however there is not general agreement about their relative importance.

Concentration polarization in crossflow MF was investigated using theoretical models by Schultz and Ripperger (1989). The following measures were suggested to reduce the concentration polarization, and to increase the permeate flux: (1) exploitation of the hydrodynamic entrance region, thereby shortening the module length; (2) installation of turbulence promoters; (3) generation of secondary flow (spiral tubes, Taylor eddy); and (4) periodic backwash.

Information From Manufacturers

Appendix A lists MF manufacturers. These manufacturers were contacted initially by telephone and then by a follow-up letter. All of the manufacturers recommend MF as a feasible option for separation of colloidal suspended solids, bacteria, and some emulsions. However, the information obtained regarding the removal of NC fines from wastewater was limited.

Samples of the RAAP poacher setting pit overflow were tested by two suppliers of crossflow membrane technology: Koch Membrane Systems, Inc., Wilmington, MA, and Millipore Corporation, Bedford, MA. Koch Membrane Systems, Inc. conducted the test using a three-cell flat cell unit with MSD-181, MSD-707, MSD-714 and MSD-717 UF membranes. They did not provide the suspended solids concentration in the concentrate stream and simply stated that UF was a feasible process. Mechanical scrubbing with sponge balls was suggested for cleaning. Fluxes, after 2 hour operation at 55 to 60 psi, ranged from 78 to 107 gallon/ft^2/day (gfd) (100 to 150 L/m^2/hr) (Balasco et al. 1987).

Millipore Corporation performed the test using PROSTAK™ module (2.0 ft^2) with 0.65 µm DVPP microporous membrane. It was reported that greater than 20 percent suspended solids was obtained in the concentrate, and the average permeate fluxes was in the range of 120 to 150 gfd at 25 °C at a feed pressure of 50 psi (Balasco et al. 1987).

Memtec, Inc. IL, markets an MF hollow fiber unit, MEMCOR,® which employs hollow polypropylene fibers with a mean pore size of 0.2 µm. The surface area of each cartridge is 1.0 m^2. The unit has a unique cleaning method. In the operating mode, the feed stream is pumped into the cartridge shell. Some liquid filters through the fiber walls and exit the cartridge as clean filtrate. The remaining feed and rejected waste flow along the fiber walls as concentrate and exits through the shell outlet. The filtrate is temporarily shut off and gas is introduced to the hollow fibers. The gas

explodes through the microporous fiber walls into the feed/concentrate stream causing violent agitation, and purges the cartridge of any waste buildup.

Osmonics, Inc. provided approximate costs for treating a 2 MGD wastewater stream using spiral type MF modules (Table 4). The labor cost given by Osmonics, Inc. was lower than that given by Arthur D. Little, Inc., while membrane replacement costs and power costs were higher. The higher membrane replacement cost might be due to the use of spiral type MF modules. Despite these variations in individual costs, overall operating and maintenance costs were almost the same.

Osmonics, Inc. and Koch Membrane Systems, Inc. indicated that it was feasible to obtain a permeate with zero NC fines but the maximum concentration would be close to 1 to 4 percent suspended solids in the concentrate. This is very different from the report made by Millipore Corporation, who claimed that it was possible to concentrate the waste stream to greater than 20 percent solids.

Because of large variation in performance, pilot studies are necessary to select the best membrane and module for removal of NC fines from wastewater. Furthermore, there is little data available on the performance of large-scale crossflow MF processes (flow rate greater than 2 MGD). Therefore, the feasibility of handling large flows by crossflow MF units must also be re-evaluated. Since past studies have shown conflicting performance results for crossflow MF systems, pilot studies are necessary to select the best membrane and module.

Table 4. Comparison of crossflow microfiltration costs.

millions/year	capital cost $ millions/year	Membrane cost $ millions/year	Labor cost $ millions/year	Power cost $ millions/year	Operating cost $ millions/year
Balasco et al.	7.79	0.34	0.09	0.15	1.99
Osmonics	5.1 to 10.2	0.51 to 1.02	0.02 to 0.04	0.23 to 0.47	0.93 to 1.86

(Note: Balasco et al. (1987) estimated costs for 5.1 MGD and Osmonics for 2.0 MGD.)

4 Laboratory Study Using Flat Sheet Crossflow Microfiltration Units

This study was conducted: (1) to investigate factors affecting the performance of flat sheet crossflow microfiltration (MF) membranes; (2) to find methods of fighting fouling and prolonging membrane life; and (3) to understand physical phenomena governing flux and explain experimental observations. Researchers from University of Wisconsin-Madison conducted this flat sheet crossflow microfiltration study.

Procedures

Table 5 lists the properties of the membranes used in the experiments. Two types of flat sheet membrane module were used. The specifications of the modules and feed pumps are provided in Table 6. The experiments were operated either in a closed loop mode or in a modified batch as discussed below.

In a closed loop mode, the flows of both the permeate and concentrated fluxes were recycled to the supply vessel; ensuring no change in suspended solids concentration. However, in a modified batch mode, the feed was supplied from a constant volume tank where the liquid level was kept unchanged by incoming fresh wastewater and by the concentrate leaving the membrane and recirculating into the system. This resulted in the increase in suspended solids concentration in the supply vessel.

Table 5. Properties of microfiltration membranes used.

Membrane Type	GVLP	HVLP	PVLP	GLSR	NMP-601
Producer	Milli-pore	Milli-pore	Milli-pore.	Amicon	Koch
Pore size, µm	0.2	0.45	0.65	0.22	1.0
Material	PVDF[1]	PVDF[1]	PVDF[1]	PS[2]	PS[2]
Configuration	Flat sheet	Flat sheet	Flat sheet	Flat sheet	Flat sheet
Comments					Research membrane

1. PVDF: polyvinylidene difluoride.
2. PS: polysulfone.

Table 6. Specifications of filtration apparatus.

Specifications	Crossflow-Plate-and-Frame Apparatus	
Type	Minitan-S	Model TM-100
Manufacture	Millipore	New Brunswick Scientific Co. Inc.
Materials	Stainless steel frame Acrylic mainfolds	Stainless steel frame Polypropylene mainfolds
Filter Area	30 cm^2	64.5 cm^2
Hold-up Volume	10 mL	2 mL
Dimensions	6"(L), 4 1/2" (W), and 3/4" (H)	7 3/4"(L), 4 1/3"(W), and 9 1/2" (H)
Pump	Cole-Parmer Pumphead: Masterflex Model 7016-20	Gear pump drive Pumphead: micropump

To perform pulsating cleaning, three solenoid values were used: one (normally open) in the concentrate line, another (normally open) in the permeate line, and the third (normally closed) in the bypass line. An automatic timer was provided to close and open these valves at required time intervals. Figure 3 shows schematic arrangement for pulsation of flux. During filtration (on), V1 and V2 were opened. V3 was close during pulsating cleaning (off). V1 and V2 were closed while V3 was opened. Figure 4 shows the details of solenoid valve operation. Table 7 shows the operational parameters used in this experimental study.

The experiments were performed with the NC-manufacturing wastewater influent of poacher pit at RAAP. The feed and permeate samples were analyzed for turbidity, total suspended solids (TSS), and pH. The procedures used to determine TSS adhered to the Temporary Procedure #L-395—Determination of TSS in NC waste established by Hercules Aerospace Inc. based on a weighing method. Turbidity was measured with the use of DRT-100B Turbidimeter (H.F. Scientific, Inc.*). TSS was found to have the following relationship at the turbidity range between 10 to 400 NTU (Nephelometric turbidity unit):

$$\mathrm{TSS(mg/L)} = 0.778\,\mathrm{Turbidity\ (NTU)} + 3.475 \qquad \text{[Eq 1]}$$

where: correlation coefficient $r^2 = 0.9995$

* H.F. Scientific, Inc., 3170-T Metro Parkway, Fort Myers, FL 33916-7597; FAX 818/332-7643, tel 813/337-2116.

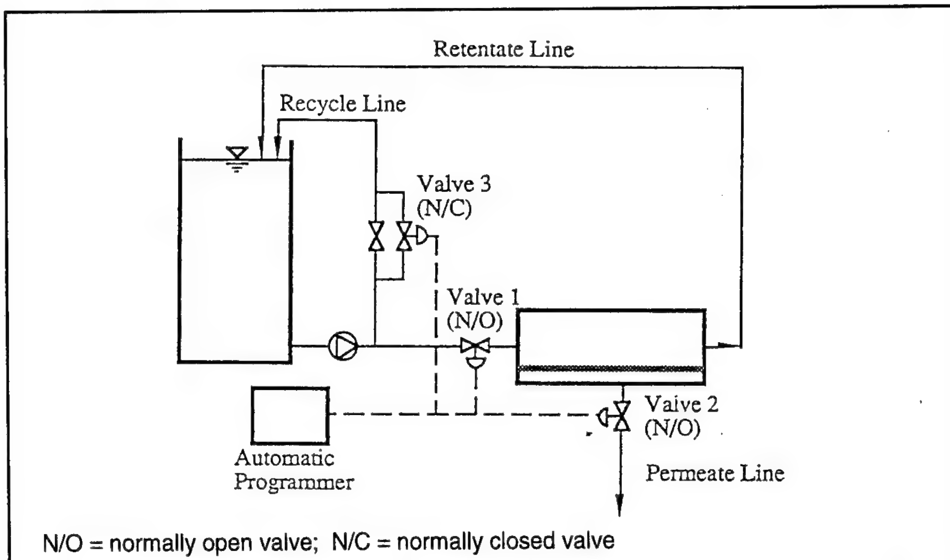

Figure 3. Arrangement for pulsating cleaning.

Another important characteristic of NC fines, which is vital in selecting MF membranes, is their average particle size distribution. A particle size analysis was performed by passing a sample through sieves and filters of different pore diameters and determining the weight of the collected material. Table 8 lists the weight distribution as a function of the particle size. This distribution is similar to the hill tank influent and poacher pit effluent (centrifuge influent) particle size distribution in Table 2. A particle size analyzer (PSA) (Brinkmann Instruments, Inc.) was also used to estimate the average size and distribution of relative sizes of the particles found in the sample based on the volume distribution. The average size of the NC particle by volume was approximately 8 μm, which was in good agreement with the results obtained from weight distribution. Note that this particle size analyzer assumed particles to be spheres. However, the majority of NC fines were long fibers. Additionally, because this analyzer cannot measure particles smaller than 0.45 μm, the NC particle size < 0.45 μm was measured by microscope. It was found from the microscopic observation that the amount of particles with a size less than 0.45 μm were negligible. The electrophoretic mobility of NC fines in a composite NC-manufacturing wastewater sample was determined using a Pen Kem Inc. System 3000 (Automatic electro-kinetic analyzer). The zeta potential calculated from the electrophoretic mobility ranged from -16.5 to -28.8 mV and the iso-electric point was approximately pH 1.9.

Factors Affecting Permeate Flux

The influence of the crossflow velocity of the fluid, u_b, in the flat sheet membrane on the permeate flux, J, was investigated at a transmembrane pressure (TMP) difference

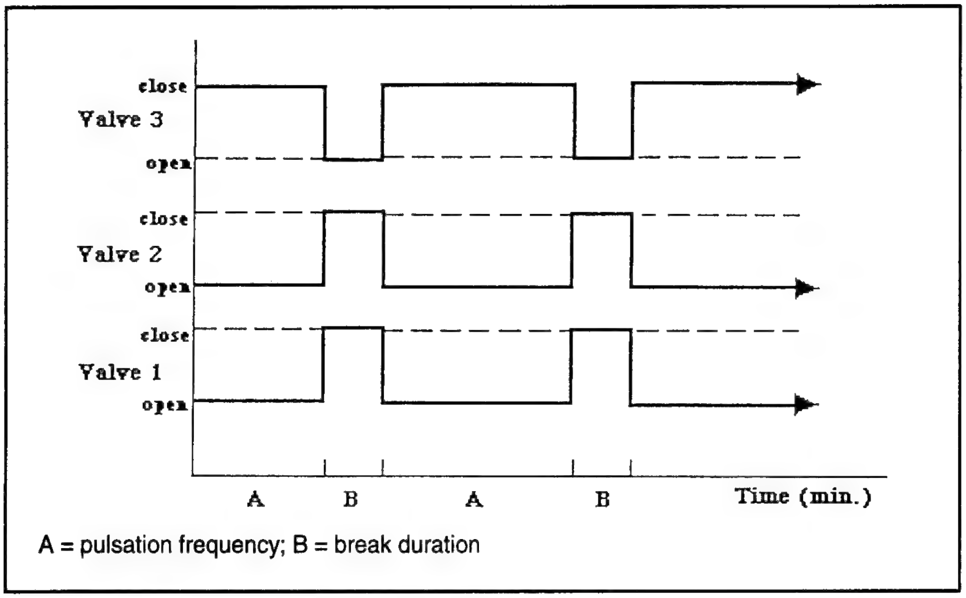

Figure 4. Mode of solenoid valve operation.

of 2 psi and a constant feedwater NC fine content (400 NTU) by the use of a closed loop operation mode (1 psi = 6.89 kPa). Figure 5 shows the changes in the permeate flux as a function of the operation time for three different crossflow velocities of 0.37, 0.82, and 1.19 m/sec. The Megaflow Membrane Filtration Apparatus (Model TM100) was used with the membrane of GLSR—Amicon, 0.45 µm.

Table 7. Experimental conditions.

Parameter	Measure
Membrane pore diameter (µm)	0.2, 0.45, 0.65, 1.0
Turbidity (NTU)	380 - 450
TSS (mg/L)	300 - 355
pH	7.5
Permeate flux (L/hr-m^2)	80 - 700
Tangential velocity (ft/sec.)	1.2 - 3.9
Transmenbrane pressure drop (psi)	0.5 - 10
Pulsating cleaning: Pulsating cycle (on/off) Pulsation breakdown (min.)	 2/1, 3/1 1/3, 1, 2
Chemical cleaning	Tergazime 0.5% or Tergazime + NaOH (pH 10)
Ambient temperature (^{0}C)	22

Table 8. Weight distribution of NC fines by size.

NC Fine Size (μm)	Weight (mg)	Weight (%)
210<	1.0	0.64
210 – 70	0.9	0.58
70 – 41	1.7	1.10
41 – 30	0.5	0.32
30 – 20	3.7	2.37
20 – 10	105.6	67.79
10 – 3	40.7	26.12
3 – 0.7	1.3	0.81
0.7 – 0.45	0.4	0.27

The increase in the crossflow velocity generally resulted in a flux increase and retardation of flux decay by reducing the cake mass deposited on the membrane surface. The deposition layer thickness in the crossflow microfiltration (MF) was controlled by the crossflow velocity as expected. The dependence of the flux on the crossflow velocity (u_b) has been known to be dominated by the flow regime in the channel (Schultz and Ripperger, 1989). The permeate flux was found to be a function of $u_b^{1/3\text{-}1/2}$. In the flat sheet membrane module used here, a laminar-flow regime generly appeared to be formed since the Reynolds number was smaller than 2320.

One of the problems in using high tangential velocity to increase the flux is the concomitant increase in the transmembrane pressure (TMP) drop, Δp, which would adversely affect the membrane performance. In addition, the reduction of cake mass deposited by increasing the crossflow velocity (u_b) diminished at high u_b values. Baker et al. (1985) found that the specific resistance increased significantly as u_b reached high values. As a result, the crossflow velocity of approximately 1.07 m/sec (3.51 ft/sec) was thought to be adequate for this application when a flat sheet module was used.

The influence of TMP drop (Δp) on the permeate flux was investigated at a constant crossflow velocity (0.82 m/sec) and a constant NC fine content of the feedwater (400 NTU). The relative flux, defined as the ratio between the actual permeate flux after a 4-hour operation time, J_a, and the initial permeate flux, J_i, was plotted as a function of Δp (Figure 6). It can be said that the relative flux decreased with the increased Δp due to the effect of membrane fouling. Initially, the increase in Δp boosted the permeate flux but accelerated the flux drop in the later phase. This flux drop can be explained by the membrane fouling resistance developed during the process filtration.

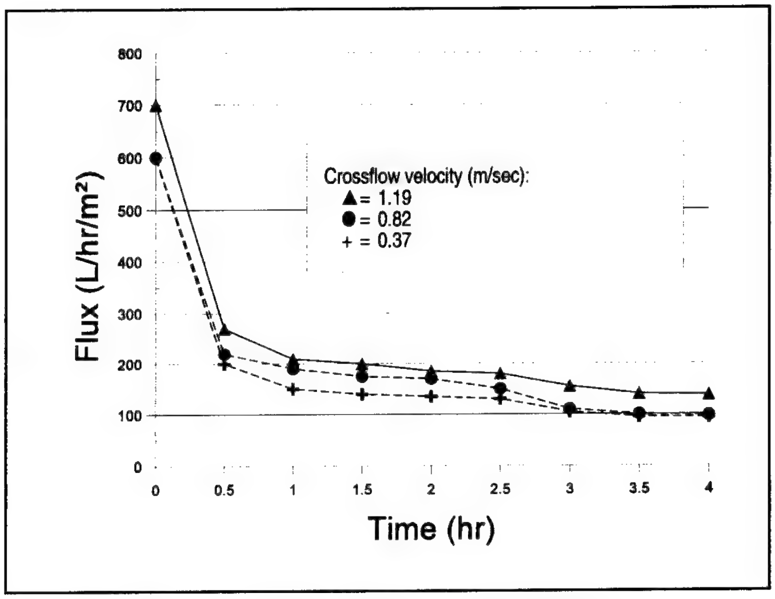

Figure 5. Effect of crossflow velocity on permeate flux.

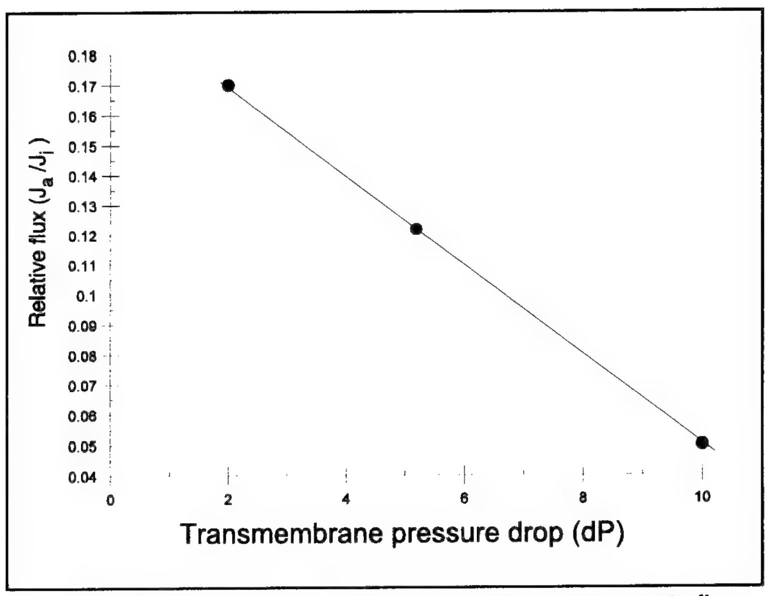

Figure 6. Effect of transmembrane pressure drop on permeate flux.

The increased Δp provides an additional driving force for higher permeate flux. Thus, particles are initially transported at a great rate to the membrane, resulting in an increased internal membrane fouling resistance and conforming to the standard blocking model (Visvanathan and Ben Acm 1989), written as:

$$J = \frac{\Delta P}{\mu (R_m + R_{if})} \qquad \text{[Eq 2]}$$

where:

J = permeate flux, m/sec

Δp = TMP drop (driving force), N/m^2

μ = viscosity of feed solution, kg/m/sec

R_m = membrane resistance, m^{-1}

R_{if} = internal membrane fouling resistance, m^{-1}

Later, when the membrane pore is sufficiently plugged with fine particles, the filtration process follows the cake filtration model, leading to the eventual formation of cake (particle deposit layer) on the membrane surface. At higher Δp, compaction of the cake could occur and the cake layer thickness increases, leading to increased external membrane fouling resistance, R_{ef}. The cake filtration model is thus given by:

$$J = \frac{\Delta P}{\mu (R_m + R_{if} + R_{ef})} \quad \text{[Eq 3]}$$

Membrane Fouling and Membrane Cleaning

Membrane fouling is the term used to describe the inorganic and/or organic deposit on the surface and consequent physical blocking of the membrane pores. Membrane fouling can cause either an increase in Δp, or decay in the permeate. Accordingly, the rise in Δp or reduction in the permeate flux indicates the progression of membrane fouling.

A series of experiments was conducted to evaluate the effect of the permeate flux on membrane fouling under the same constant crossflow velocity using a modified batch operation mode. The Minitan-S tangential-flow filtration unit (Millipore Co.*) was used with an HVLP membrane.

Figure 7 shows the TMP drop as a function of operation time for three various permeate fluxes. It was apparent that maintaining higher permeate flux resulted in sharper increase in TMP drop during the first hour, indicating accelerated membrane fouling. A higher permeate flux yielded transport of NC fines at a greater rate to the membrane. Under the higher permeate flux condition, the thicker retained layer composed of densely packed NC fines was formed on the membrane surface. Also, pore blockage had apparently occurred before the formation of a thicker retained layer. MF

* Millipore Co., 80-T Ashby Rd., Bedford, MA 01730, tel. 617/275-9200.

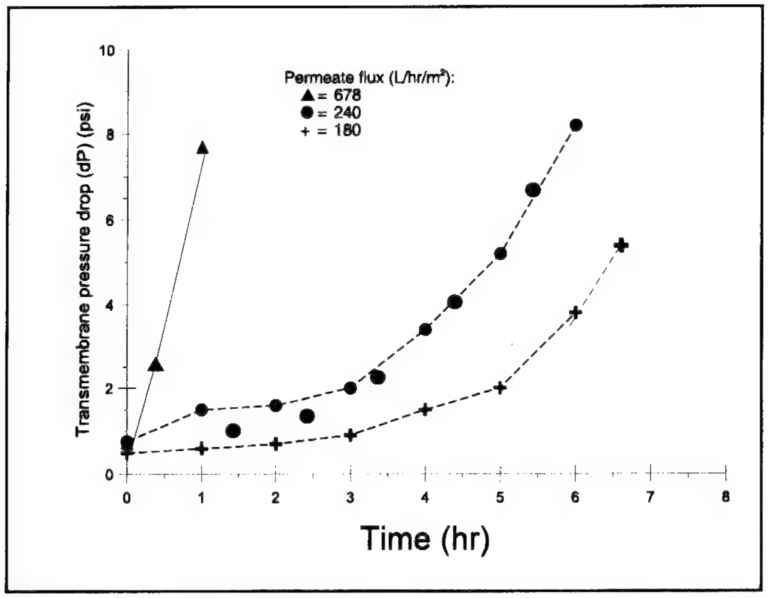

Figure 7. Effect of permeate flux on transmembrane pressure drop.

membrane pores were susceptible to blockage by NC fines due to the wide range of NC fine particle size distribution, and the rod-shaped, soft, flexible NC fines. To avoid premature membrane fouling, it is recommended to operate the crossflow MF in a modest permeate flux (100 to 150 L/hr/m^2).

The effect of membrane pore diameter on filtrate turbidity was studied at a constant NC fine concentration in the feedwater over a 4-hour running period using a closed-loop operation mode. The results (Table 9) show that the difference in the filtrate turbidity among the various pore size membranes was marginal, especially after 0.5 hours of operation.

Another set of experiments used three different pore diameters: 0.2, 0.45, and 0.65 μm under a modified batch operation. The permeate flux was kept constant by increasing the TMP during the operation. For all pores, the turbidity of the filtrate observed was almost zero. Even in the initial time period it was far below the discharge limit for NC-manufacturing effluent of 40 mg/L. All the MF membranes exhibited an initial period of filtration in which more NC fines appeared in the filtrate. After this period, the amount of NC fines in the membrane decreased and the permeate flux decayed.

It is desirable to use the largest possible pore diameter to maximize the MF permeate flux. The membrane of 0.6 μm pore diameter is recommended for the flat sheet membrane microfilters used here. (This conclusion is to some extent restricted by the current limited commercial availability of larger pore diameter membranes.)

Table 9. Variation of filtrate turbidity over time using different pore size membranes.

Time (hr)	Turbidity (NTU) 1.0 µm	0.65 µm	0.45 µm	0.2 µm
0.0	2.30	2.10	2.10	0.70
0.5	0.40	0.40	0.35	0.17
1.0	0.18	0.21	0.20	0.12
1.5	0.20	0.19	0.17	0.12
2.0	0.16	0.17	0.16	0.12
2.5	—	0.18	0.16	0.11
3.0	0.16	0.17	0.16	0.10
3.5	—	0.17	0.15	0.11
4.0	0.17	0.16	0.15	0.10

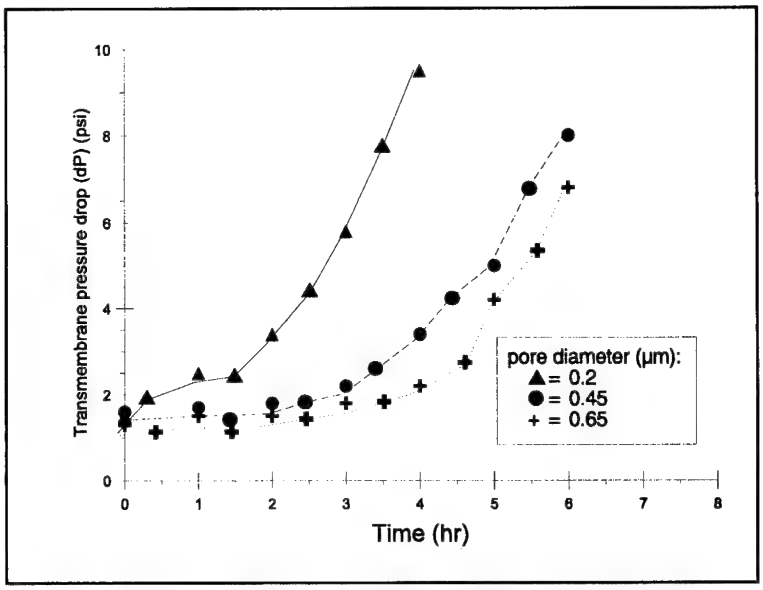

Figure 8. Effect of pore size on transmembrane pressure drop vs. time.

Figure 8 shows that the TMP drop (Δp) increased over time for all these membranes. The membrane with smaller pore (0.2 µm) however, experienced a much higher drop in TMP at a given time. Figure 9 shows that Δp rose sharply as the feedwater was concentrated. Smaller pore membrane appeared to be more sensitive to increased NC fine concentration. The membrane with 0.2 µm pore size showed a much higher rate of Δp increase (7 psi in 4 hours) as the final concentration reached 6 times the initial concentration. However, Δp increase for the 0.65 mm pore diameter membrane was 6 psi in 6 hours as the final concentration reached 8.5 times the initial concentration.

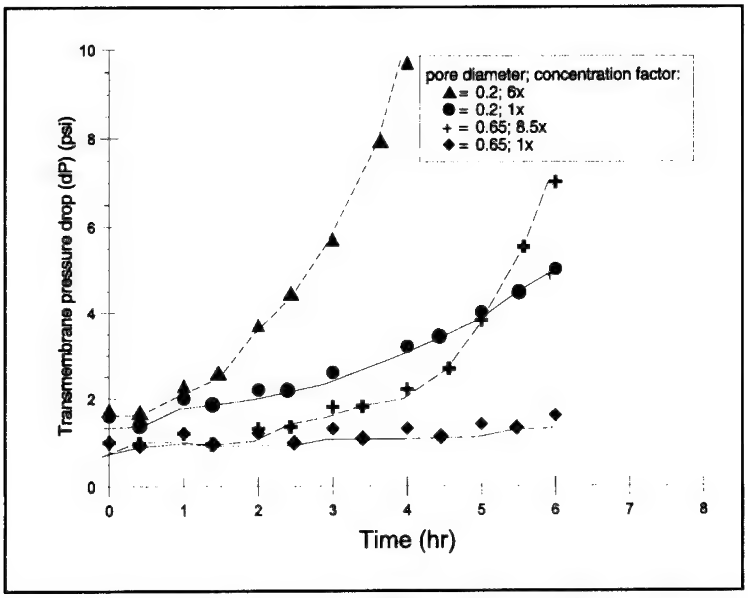

Figure 9. Effect of feedwater turbidity on transmembrane pressure drop vs. time.

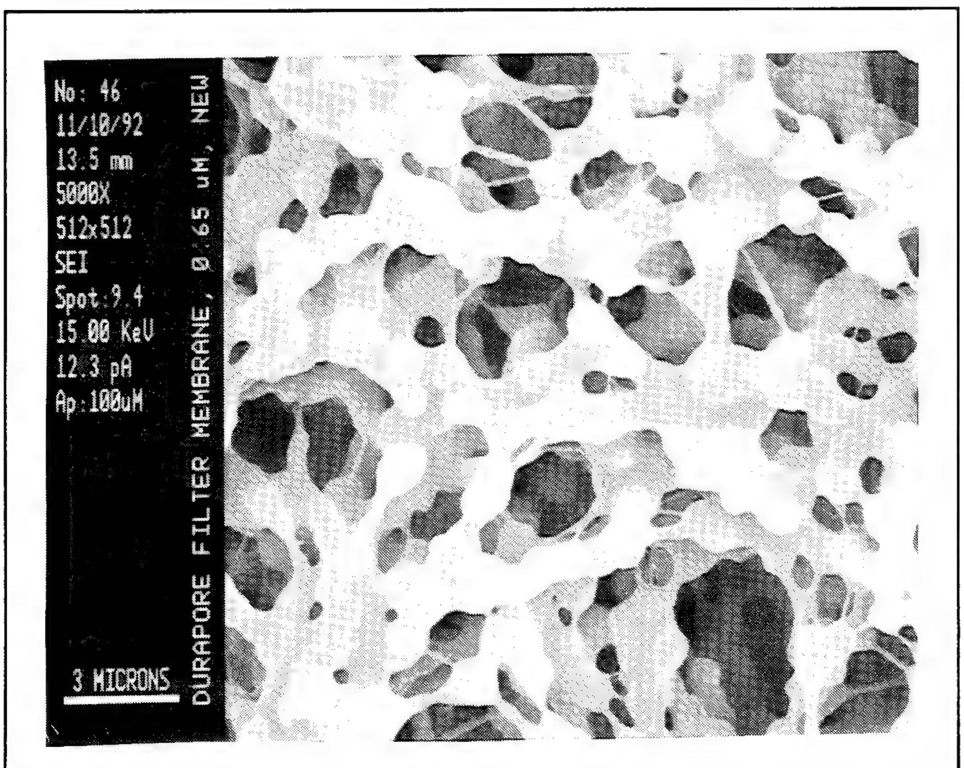

Figure 10. SEM picture of top layer of MF membrane, type PVLP, Durapore (Millipore) (0.65 μm).

The increased NC fine concentration of the feedwater created a higher membrane fouling rate. The increased turbidity and thus increased viscosity of feed solution in general would result in the deterioration of the permeate flux. The higher membrane

fouling rate due to the increased turbidity of the feedwater shown in this experiment was most likely related to this flux deterioration.

Scanning electron microscopy was used to characterize fouled membranes, as well as the structure of MF membranes. Figures 10 and 11 show the top views of the MF-Millipore (0.65 µm) and MF-Amicon (0.45 µm). The membranes had high porosity, rough surfaces, and a sponge-like and tortuously microporous structure.

Membrane fouling was examined after a long-term run when the TMP difference had risen to over 10 psi. Figure 12 shows a cross-section of the top cake layer formed on the membrane, which has a thickness of about 80 µm. Figure 13 shows the top view of the surface of the fouled membrane with further magnification. It can be seen that the cake layer is composed of densely packed debris of NC fines.

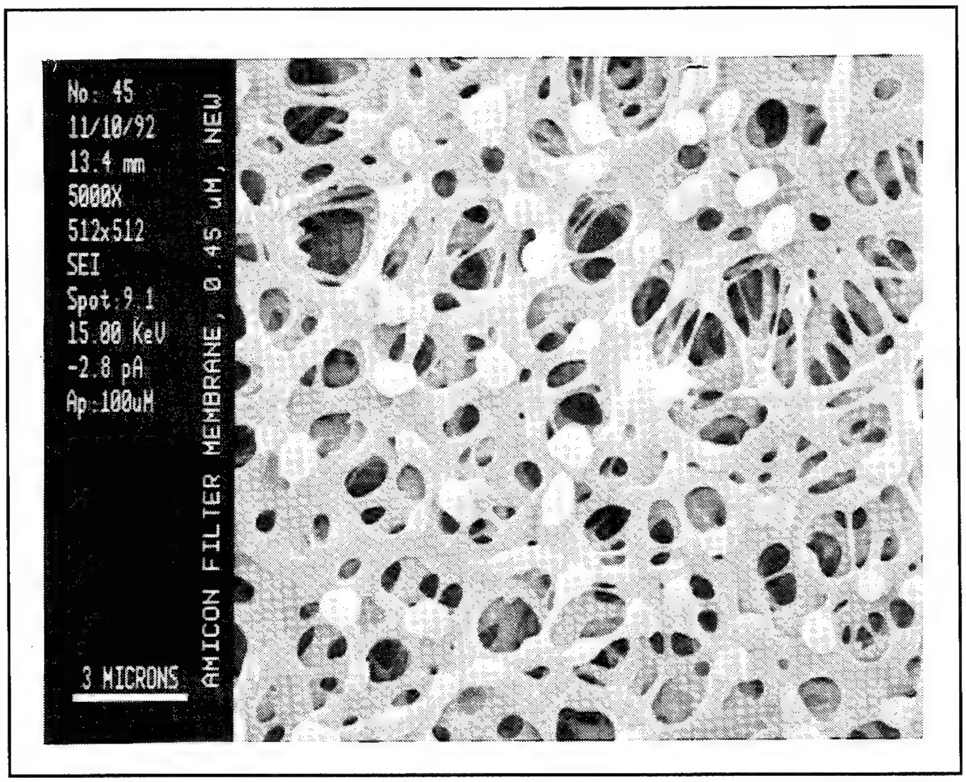

Figure 11. SEM picture of top layer of MF membrane, type GSLR, Amicon (0.45 µm).

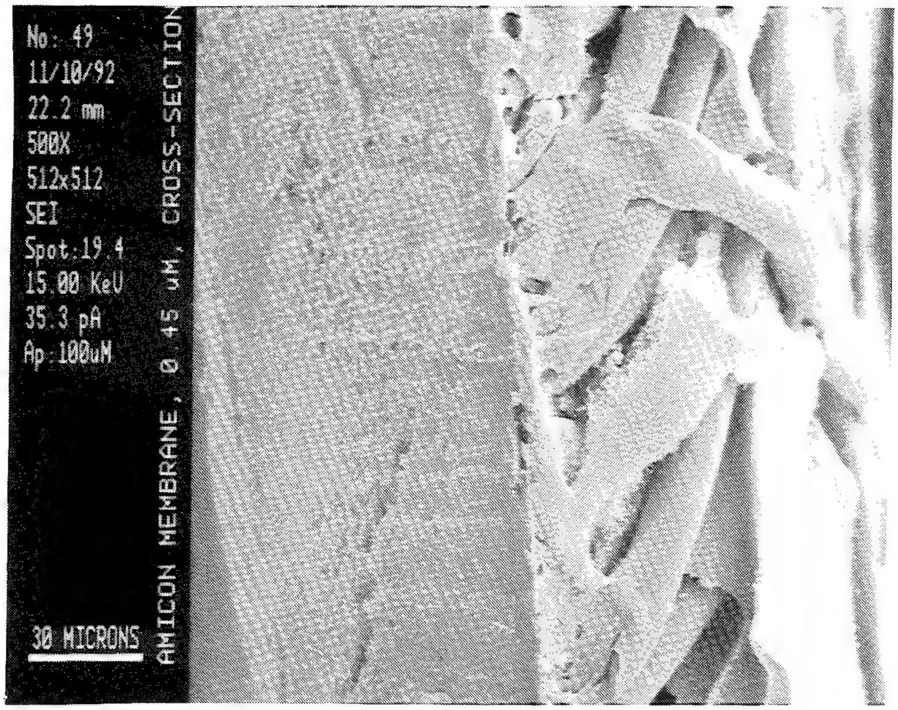

Figure 12. SEM micrograph of cross-sectional view of NC fines deposit layer and fouled membrane.

Deposition of particles of different shapes and sizes on the top surface was examined. Figure 14 shows that particles smaller than the size of pores were trapped in the pores, bridging the surface pores of the opening. Pulsating and chemical cleanings were attempted to combat membrane fouling.

The pulsating cleaning technique was designed to minimize membrane fouling developed during the MF of the NC-manufacturing wastewater. Shock waves were created through the membrane by closing and opening the valves at required time intervals, which were controlled by an automatic timer. The operation cycle used during the experiment was defined as time on/time off (Figures 3 and 4). The experiment was conducted with different cycles and different break durations (off).

The permeate flux was improved when using pulsating cleaning compared with no pulsating cleaning based on the actual working hours under the similar operation condition. The flux increased by 50 percent during a 7-hour operation when the running cycle of the on/off ratio of 2:1 with off-time of 2 minutes was applied. The permeate flux behaviors as a result of pulsation (Figure 15) suggest that the longer break duration would result in better improvement. In fact, pulsating cleaning

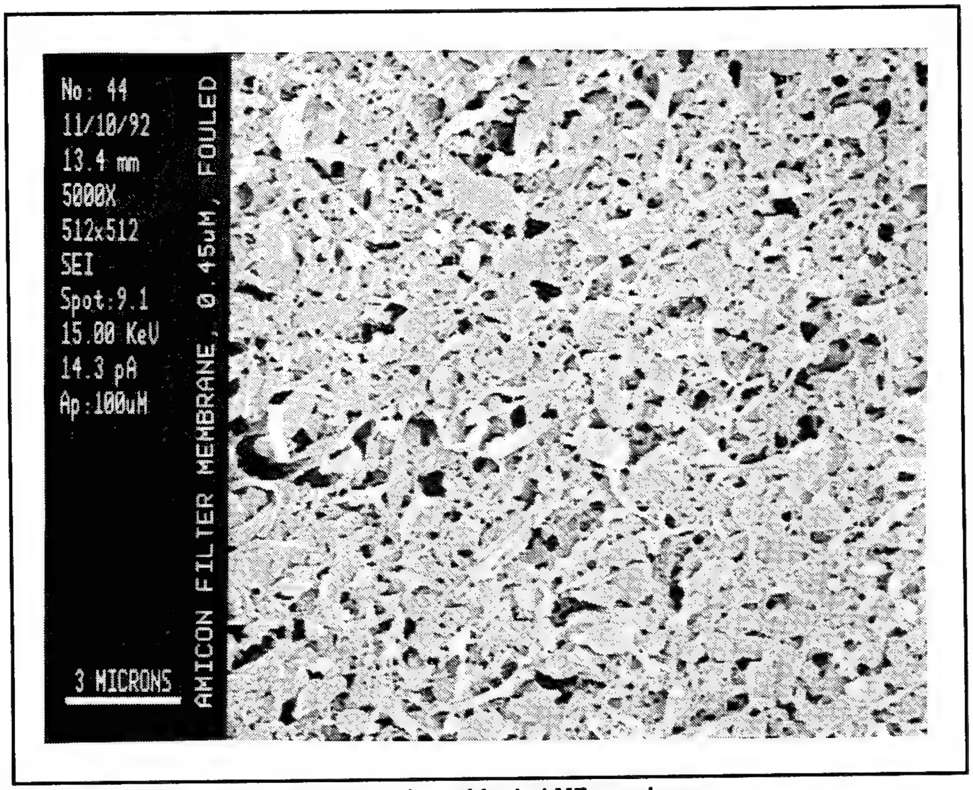

Figure 13. SEM micrograph of top view of fouled MF membrane.

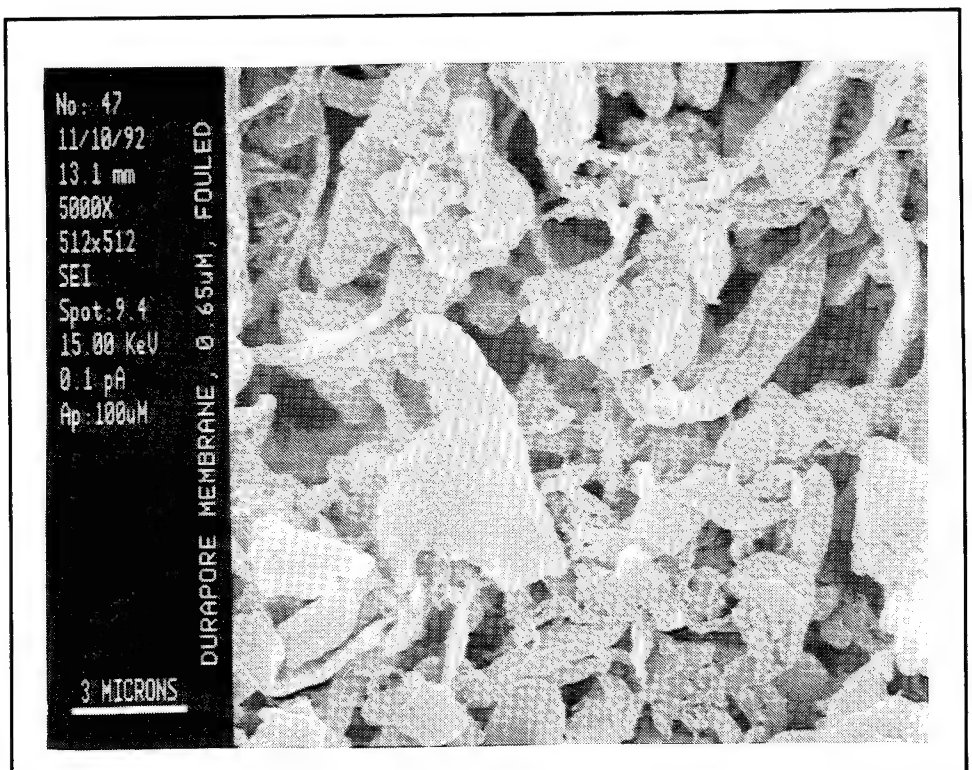

Figure 14. SEM picture of deposition of NC fines on top layer of MF membrane.

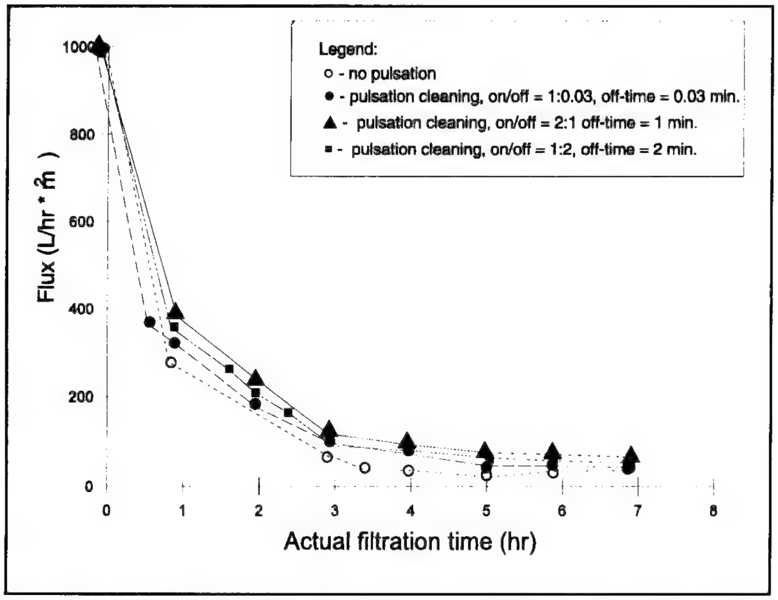

Figure 15. Effect of feedwater turbidity on transmembrane pressure drop vs. time.

operation worked as self-cleaning, which is an important measure to achieve long-term practical flux level for the MF modules such as in a flat sheet membrane module where backflush cannot be used due to the non-self-supporting structure of the membrane.

Chemical cleaning of the fouled membrane was attempted by removing membrane foulants using specific chemical agents. Membranes were cleaned chemically when the TMP drop was greater than 10 psi. Pure water fluxes measured before and after cleaning were used as indicators of degree of cleaning. Different kinds of cleaning solutions were stirred in contact with the fouled membrane in a vessel at room temperature (22 °C) for 5 hours to simulate a generally accepted clean-in-place procedure. Then the membrane was rinsed and flushed with pure water for approximately 15 minutes to wash out all the absorbed chemical agents.

It was found that 0.5 percent Tergazyme® solution* by weight cleaned the fouled membrane best for the MF membrane types GVLP, HVLP, and DVLP, and 0.5 percent by weight Tergazyme solution plus NaOH to adjust pH to 10 for GLSR. The flux after cleanup achieved almost 90 to 100 percent of the initial pure water permeate flux.

Transport Mechanism

Viewing the complex phenomena of the permeate flux decline and membrane fouling shown in the present experimental studies provided a motivation to explore theoretical

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* Tergazyme is a registered trademark belonging to Alconox, Inc., 9 E. 40th St., Suite 200, New York, NY 10016-0402, tel. 212/532-4040.

models to understand and describe the transport mechanism governing the filtrate flux. To date, very few models proposed for filtrate flux have been considered adequate to explain experimental observations or predict device performance (Gekas and Hallström 1990).

In the earliest work in this area, crossflow MF was regarded as a process where the carrier liquid was removed through porous medium using pressure as the driving force and particulates deposited on the separating medium. The cake filtration theory was applicable to a certain degree but obviously limited since shearing force caused by tangential flow was not considered. Other attempts included the application of the model used in the ultrafiltration process, which was based on a concentration polarization concept where the drag force was balanced at steady-state by Brownian diffusivity of the particles. Application of this kind of model, however, has proven to give significant discrepancy between the prediction of the model and experimental results.

The ultrafiltration model underestimates the filtrate flux of crossflow MF, as much as two orders of magnitude lower than that of actual observation, because the value of the Brownian diffusion coefficient, which is inversely proportional to the particle radius, is quite small even for micrometer-sized particles. Various models were developed to modify the ultrafiltration model, such as the model based on the theory of shearing-induced hydrodynamic diffusion arising from particle-particle hydrodynamic interaction. Zedney and Colton (1986) proposed the following model to incorporate shear-enhanced diffusivity of the large floc:

$$J = 0.078\left(\frac{a^4}{L}\right)^{\frac{1}{3}} \gamma_w \ln\left(\frac{C_w}{C_b}\right) \qquad \text{[Eq 4]}$$

where:

J	=	length average flux, cm/sec
a	=	particle radius, cm
γ_w	=	wall shear rate, sec^{-1}
C_w	=	particle content at wall, dimensionless
C_b	=	particle content in bulk solution, dimensionless
L	=	channel length, cm

Zedney and Colton also claimed that the agreement with experiments was good for membrane plasmapheresis; however, the model did not include the effects of particle deformability and the hydraulic resistance of the particle layer at the membrane.

Besides, it is impracticable to define particle radius in the case where a wide range of particle size distribution exists.

It was thought that the model for crossflow MF used in separation of particle suspensions could be a hybrid somewhere between basic filtration and ultrafiltration, which incorporates the concepts of diffusivity (back transport) and the known equation of the filtration theory such as [Eq 3]. For an approximation, the membrane resistance and internal resistance, which are relatively insignificant in comparison with the external resistance under a steady or a quasi-steady operation, can be neglected; thus [Eq 3] becomes:

$$J = \frac{\Delta p}{\mu R_{ef}} \qquad \text{[Eq 5]}$$

Since the resistance of the particle deposit is proportional to its thickness, δ, and the specific resistance of the layer, r, R_{ef} may be defined as:

$$R_{ef} = r\,\delta \qquad \text{[Eq 6]}$$

hence,

$$J = \frac{\Delta p}{\mu\, r\, \delta} \qquad \text{[Eq 7]}$$

Under the steady-state condition, the retained suspended particles transported towards the membrane (convection) is balanced by the material of the suspended particles transported back into the bulk stream because of shear-induced hydrodynamic diffusion. On the basis of mass balance the transport rate by convection can be described as:

$$m = J \frac{\rho}{\rho - C_s} C_s \qquad \text{[Eq 8]}$$

where:

m	=	mean flow of the retained material, $kg/m^2/sec$
ρ	=	density of particle deposit layer, kg/m^3
C_s	=	concentration of particle in bulk stream, kg/m^3

For the back-transport, it is assumed that this rate is proportional to velocity gradient, du/dy on the membrane and cake-layer thickness, δ:

$$m' = K \delta \frac{du}{dy} \rho \qquad \text{[Eq 9]}$$

here:

m' = mean flow of back-transported material, $kg/m^2/sec$.

The mass balance for steady-state can be expressed as:

$$J \frac{\rho}{\rho - C_s} C_s = K \delta \frac{du}{dy} \rho \qquad \text{[Eq 10]}$$

For the Newtonian fluid, velocity gradient, du/dy, is found to be a function of shear stress, τ, and fluid viscosity, μ:

$$\frac{du}{dy} \sim \frac{\tau}{\mu} \qquad \text{[Eq 11]}$$

In the case of laminar flow, shear stress is not only proportional to the tangential velocity, u_b, but also depends on μ and the channel half-height, h:

$$\tau = 3 \frac{\mu}{h} U_b \qquad \text{[Eq 12]}$$

Equations [7] and [12] lead to the following equation for the permeate flux:

$$J = \left[\frac{K \Delta p \, u_b \, (\rho - C_s)}{\mu \, r \, h \, C_s} \right]^{\frac{1}{2}} \qquad \text{[Eq 13]}$$

where :

J = permeate flux, m/sec

Δp = transmembrane pressure drop, Pa

ρ = density of particle deposit layer, kg/m^3

u_b = tangential fluid velocity, m/sec

C_s = concentration of particle in bulk stream, kg/m^3

μ = dynamic viscosity of fluid, Pa · sec
r = specific deposit layer resistance, m^{-2}
h = half-height of flow channel, m
K = dimensionless constant.

A similar model based on filtration theory has been proposed by Schultz and Ripperger (1989). [Eq 13] was derived for flat sheet microfilter operated in a lower Reynolds number (< 2320).

In crossflow MF, membrane fouling and concomitant flux decline strongly depending on the physical property of particles, membrane morphology, and the interaction between particles and membrane. It is difficult to define real pores and smooth geometric surface at the micrometric level. The situation is complicated by the wide range of particle size distribution and the peculiar characteristics of NC fines such as shape and deformibility. Nevertheless, fouling leads to an increase of resistance to permeate by thickening and/or compression of the particle deposit layer, which are included in [Eq 13] as r, specific resistance. It is clear from [Eq 13] that the TMP drop and crossflow velocity at steady state are reduced to a certain extent by the tendency of a particle deposit layer of increased high specific resistance to form. Obviously, if the particle causing the blockage and the formation of a cake layer did not limit the permeate flux, the flux should increase with an increased TMP drop, contrary to the experimental results. In addition, specific resistance depends on the crossflow velocity. The effect of crossflow was diminished at a high u_b value due to the significant increase of specific cake resistance arising from the exclusion of large particles from the cake (Baker et al. 1985; Rushton and Zhang 1988).

The permeate flux is related to the channel half-height, h, which would be decreased due to the particle deposition and the build-up of cake thickness. The decrease causes an increase in shear stress since the same volume of suspension must flow through a more restricted channel, leading to an increase in diffusivity, which then balances the rate of particle deposition on the layer. Variation in the particle concentration, C_s and the viscosity of fluid, which is a strong function of C_s, will complicate the dynamic equilibrium. This effect is incorporated in [Eq 13]. Davis and Birdsell (1987) showed that the cake layer thickness increased with particle concentration of bulk stream. Adverse influence of increased C_s on membrane performance were also observed in the experiments mentioned above.

In summary, this model was found to adequately explain the foregoing experimental observation. The model incorporating shear-induced hydraulic diffusion and cake filtration theory is expressed in terms of macroscopic properties such as tangential fluid velocity, TMP drop, specific resistance, fluid viscosity, and permeate flux, all of

which are evaluated from the experimental measurements. One must know the value of specific resistance to predict permeate performance. A range of 1×10^{16} to 1×10^{17} m^{-2} of r in steady-state or quasi steady-state operation was calculated in this experiment. Further development and validation will be performed in the future.

5 Field Evaluation Using a Hollow Fiber Crossflow Microfiltration Unit

The objective of this study was to evaluate whether a hollow fiber crossflow microfiltration (MF) process could be used to replace or upgrade the existing DeLaval centrifuges removing NC fines from wastewater at RAAP. The following questions were addressed: (1) how effectively can NC fines be removed from wastewater and concentrated, (2) what are the optimized process parameters for an extended period, and (3) what is the optimum cleaning procedure (in terms of agents, frequency, and economics). In addition, design criteria information was developed and preliminary economic analysis was performed. (Researchers from Hercules Aerospace Inc., RAAP, conducted this hollow fiber crossflow MF study.)

Procedures

Assessment of DeLaval Centrifuges Capability

Assessment of the NC fines removal capabilities of the DeLaval centrifuges was necessary before selecting MF equipment. DeLaval centrifuge performance data were previously collected to show compliance with state-mandated effluent guidelines. From 19 October 1990 to 14 December 1990, the concentration of total suspended solids (TSS) (mg/L) in the influent to the centrifuges averaged 407.8 ± 121.9 vs the effluent concentration of 74.1 ± 27.9, indicating a removal efficiency of 82 percent.

Screening of Large NC Particles

The vendor for the MF equipment requested that a series of experiments be performed to determine the screen size (mesh) required to protect the MF modules from NC particles larger than 100 μm. Therefore, samples of poacher pit influent were passed through screens of 150, 200, and 250 mesh to determine the optimal screen size to protect the MF membranes from larger NC particles. The resulting filtrate was examined using the optical microscope. Table 10 lists NC particle length and diameter determined during these tests as a function of screen size. These experiments showed that the unscreened sample contained NC that was greater in length than 100 μm, while all three screened samples effectively removed particles greater than 65 μm in

length. Therefore, a 200-mesh (subsequently referred to as 100 μm) screen was selected.

Table 10. Determination of screen size required to remove NC fibers greater than 100 µm

	Sample	
Largest Particle	Length, µm	Width, µm
Unscreened sample	146	65
150 mesh screen	49	26
200 mesh screen	65	32
250 mesh screen	65	20

Description of the Pilot Memtec MF Unit

Memtec MF uses microporous hollow fibers encased in modules to provide high surface area for filtration. The actual filtration modules used in this evaluation employed hollow polypropylene (PP) tubes with an internal lumen diameter of approximately 350 μm and external diameter of 650 μm. The wall thickness of these tubes is approximately 150 μm and the average pore size is 0.2 μm. Each of the modules contains 3000 of these hollow fibers to provide a filter surface area of 1 m^2 per module. Figure 16 shows the Memtec MF unit and Figure 17 shows a flow sheet diagram of the unit. It should be pointed out that the system had been modified to reflect safety requirements. Modifications included using pedestal type sump pump and MEMA-4 electrical rating motors and other components.

During operation, liquids to be filtered are pumped through the modules under moderate pressures (approximately 20 psi). The lumens of the hollow fibers are not pressurized and thus a pressure differential is created. This pressure across the membrane is referred to as transmembrane pressure (TMP), which is the driving force for filtration. Since the membranes have 0.2 μm pores, the solids are retained on the outside of the fibers. The concentrated solids are then periodically collected from the crossflow loop during backwash. The filtered water in the lumens is continuously released as clean effluent.

A unique feature of the Memtec technology is the patented gas backwash system, a preprogrammed microprocessor system. This system periodically introduces air under high pressure into the center (lumen) of the hollow fibers to remove trapped contaminants and accumulated solids from the surfaces of the hollow fibers. The manufacturer claims that the backwash system provides the ability to achieve higher flow rates for extended periods of time when compared to traditional MF methods. Another feature of the Memtec system that increases filter efficiency is the crossflow configuration. In this configuration, the liquid to be filtered is continuously swept past the membrane surfaces, thus reducing the accumulation of particles on the surface and concentrating the NC in the crossflow loop.

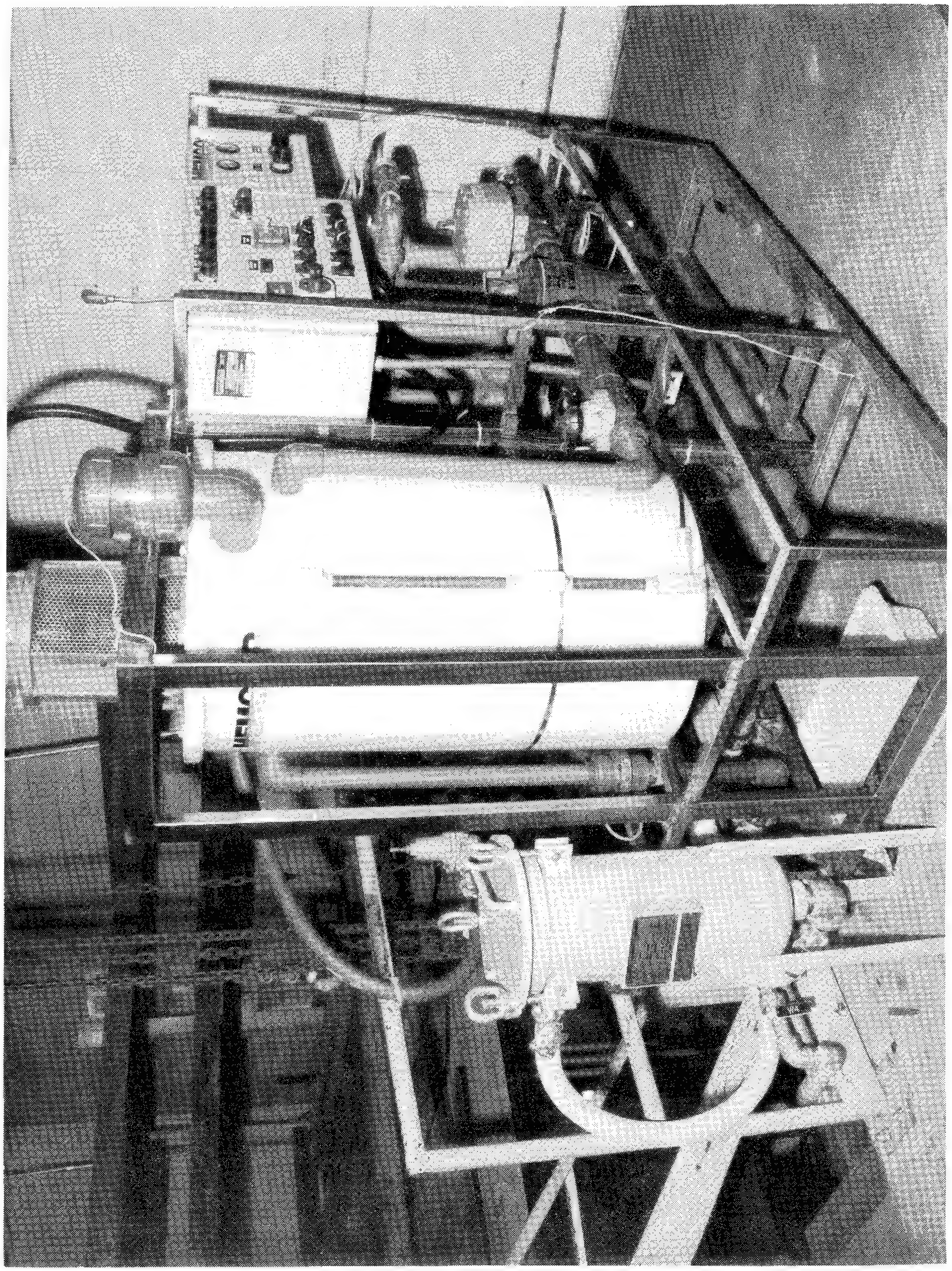

Figure 16. Front and left side view of microfiltration unit.

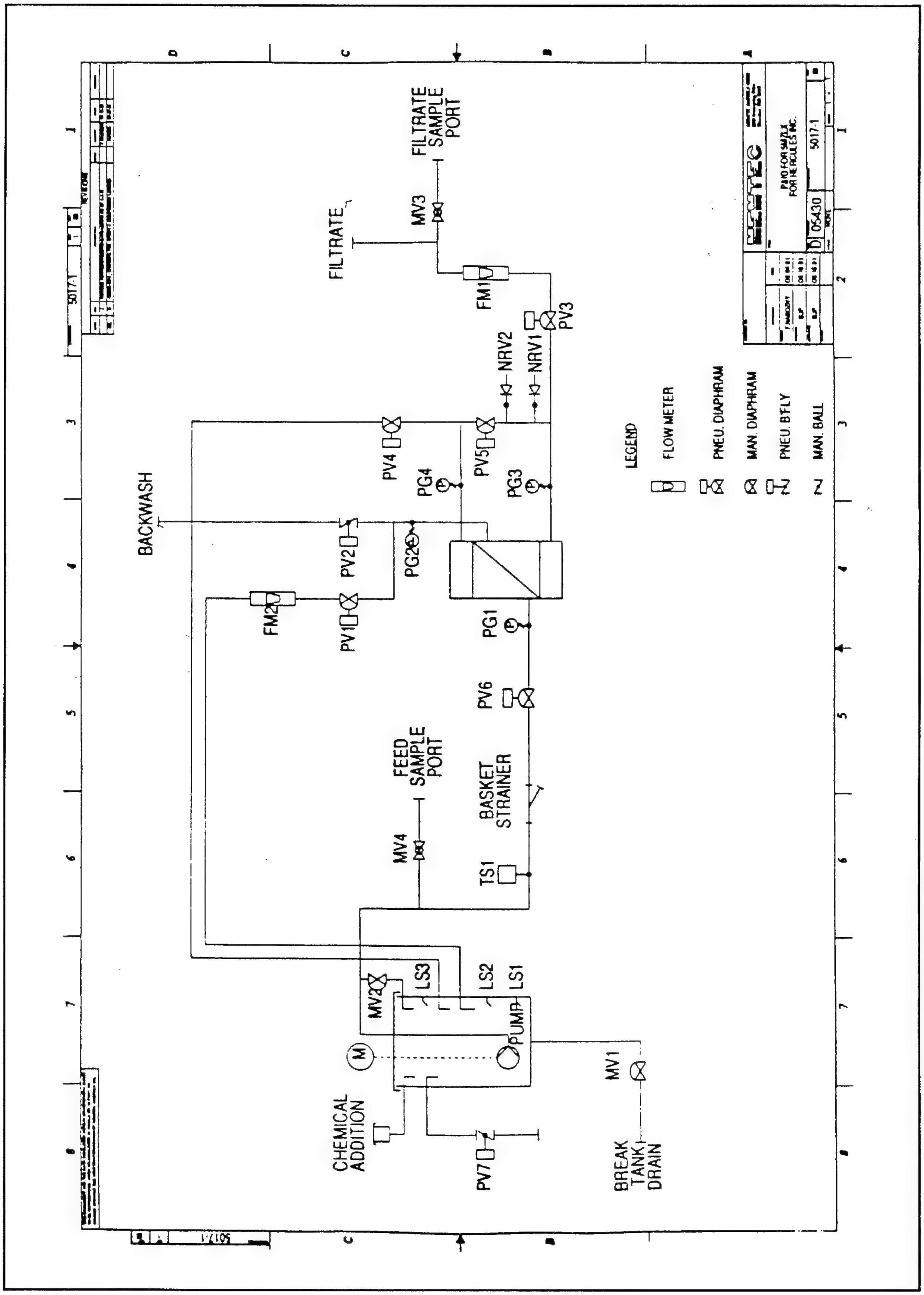

Figure 17. Process flowsheet for microfiltration unit.

Membrane or filter pore size is typically established based on the particle size of the material to be filtered, desired flux rates, mechanical limitations, pressure constraints, and TSS requirements of the filtrate. These elements were considerations in determining the membrane pore size used by Memtec; however, the unique properties of the gas backwash system bear remarkably on the choice of pore size. Backwash of the 0.2-μm pore size filters can be accomplished with pressures of 100 psi. A smaller pore size would require greater backwash pressures which would exceed the stress capabilities of the construction materials.

Equipment Installation

Building 3025 in C-line, RAAP, was chosen as the installation site based on its proximity to the poacher pit and DeLaval centrifuge buildings, isolation from other processes, provision of shelter, and collection and confinement capabilities in the event of an NC spill. Preliminary equipment drawings were provided by Memtec and installation sketches were prepared.

The unit was installed and Memtec personnel were present to observe the water testing and initial processing of wastewater-containing NC fines. No significant operational problems were encountered.

Test Plan

A test plan containing three different Phases (Water, Site, and Extended Operation) was developed to evaluate the pilot equipment. Memtec engineers reviewed the proposed test plan and recommended several modifications to improve evaluation of the equipment. One revision involved extension of the run period for each test to permit solids (fines) concentration to reach equilibrium in the MF crossflow loop. Table B1, Appendix B, summarizes the modified test plan.

Results and Analysis

Table B2, Appendix B, presents an example of the experimental data on the pump operating time, backwash timer setting, pressure readings from the four crossflow pressure gages (Figure 17), TMP, crossflow rate and filtrate flux flow rate. Memtec recommended that a 1 psi correction factor be added to all the TMP values to compensate for pressure gauge error (TMP data recorded herein have not been corrected). These data along with the removal efficiency of NC fines are discussed in detail in the following sections.

Phase 1, Water Test

The water test consisted of five evaluations performed both at the vendor location before acceptance (Tests 1 through 3), and after installation at RAAP (Tests 4 and 5). Operation time (15, 30, and 60 min) and filtrate flux (2.5, 5, and 10 gpm) were the primary variables investigated. No problems were encountered with the Memtec unit.

Phase 2A, Site Evaluation Tests

Phase 2A tests were performed on DeLaval centrifuge effluent since it contains the lowest concentration of TSS. The DeLaval centrifuge effluent had an average TSS of 30 ± 13 mg/L. In all, 9 tests were performed on the DeLaval centrifuge effluent. The MF unit was operated for 240 min for each test condition to ensure equilibrium. Filtrate rate from the unit was varied (1, 2, 2.5, 3, 4, 5 gal/min) and backwash frequency was varied (10, 15, 20, 30, 60 min intervals) (1 gal = 3.78 L). In tests 6 through 10, the filtrate rate was varied while maintaining a constant backwash frequency.

Figure 18 shows a plot of TMP vs operating time at a constant backwash frequency of 15 minutes for the six different filtrate fluxes. This figure demonstrates the effects of varying filtrate flux. Under these operational parameters, it appears that filtrate fluxes of 1, 2, 2.5 and 3 gpm are similar. However, when the filtrate flux is increased to 4 or more gpm, the trend line for the TMP increases as a function of time. At higher filtrate fluxes, the backwash at 15 minute intervals is apparently no longer able to

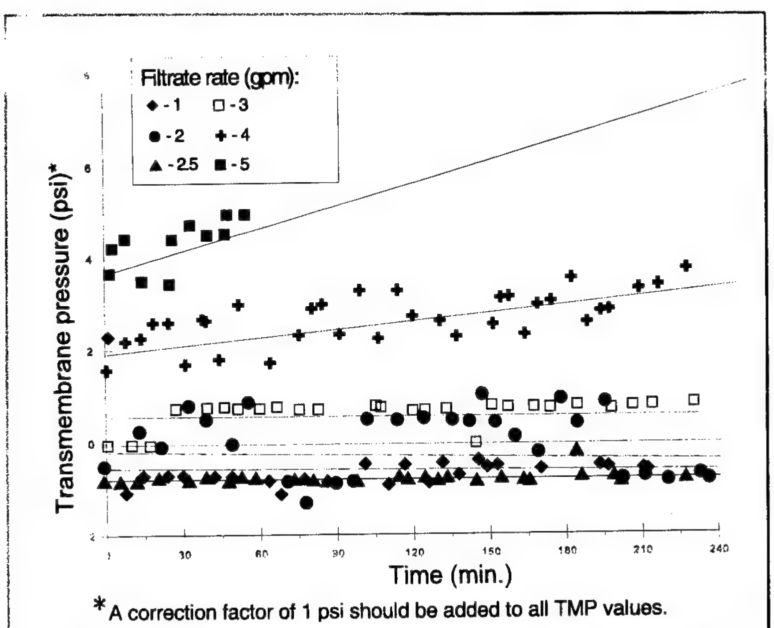

Figure 18. Transmembrane pressure vs. time with constant (15 min) backwash for centrifuge effluent.

completely clean the membranes and, therefore, the membrane efficiency decreases as a function of time. Optimal filtrate rate was determined to be 4 gpm. A similar series of tests were performed with the filtrate flow rate held constant (4 gpm) while the backwash was varied (tests 10, 12, 14, 15, and 16). However, results from these tests indicated that the MF unit would require backwashing only once per hour.

Table 11 shows the TSS data obtained during tests 6 through 16. The solids concentrations, after 240 minutes of operation, are plotted in Figures 19 and 20. Figure 19 demonstrates that increasing filtrate rate from 1 to 4 gpm, and with a constant backwash frequency of 15 minutes, resulted in an increase in solids concentration from 240 mg/L to 742 mg/L. The trend line for the data (Figure 19) shows that increasing the filtrate rate from 1 to 4 gpm increased solids concentration in the backwash from 350 to 1025 mg/L. Figure 20 shows the effects of backwash frequency on NC fines concentration. As the interval between backwashes is increased from 10 to 60 min, the solids concentration increases from 512 to 3360 mg/L.

Table 11. Microfiltration TSS concentration utilizing DeLaval centrifuge effluent.

Time Min.	Test No.	Filtrate Rate (gpm)	Backwash Freqency (min.)	TSS (mg/L)			
				Influent	Crossflow	Filtrate	Backwash
120	6	1	15	47	74	4.5	223
240				35	71	0.1	240
120	7	2	15	44	16	0.4	312
240				6	26	0	470
120	8	2.5	15	26.5	46	5.5	726
240				24.5	59.5	5	891
120	9	3	15	46	82.5	2.5	854
240				58	93.5	3.5	1145
120	10	4	15	12	23	0.8	93
240				24	48	1.6	742
120	12	4	10	31	43	1.1	844
240				31	53	2	512
120	14	4	20	37	50	1.4	1072
240				38	54	1	1244
120	15	4	30	20	49	10.1	1872
240				20	47	0.2	1504
120	16	4	60	18	39	0	4340
240				34	45	0	3360

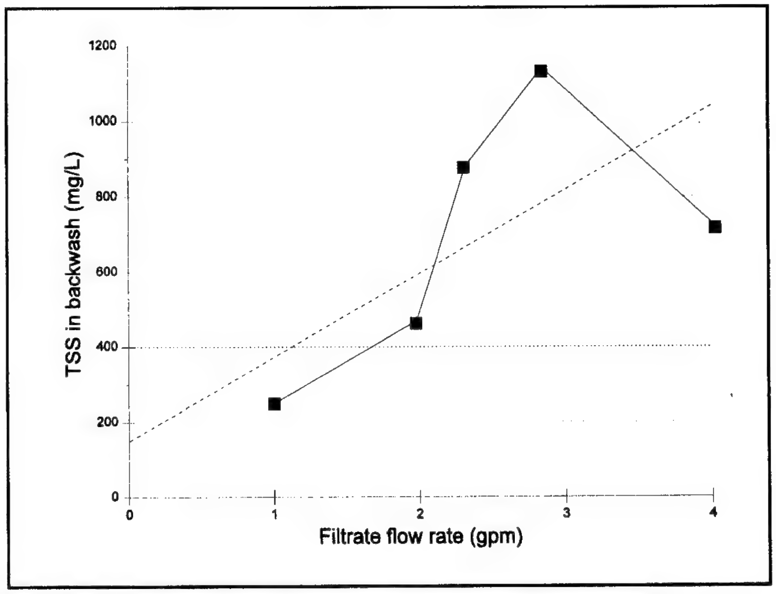

Figure 19. TSS vs. filtrate flow rate with constant backwash frequency (15 min.).

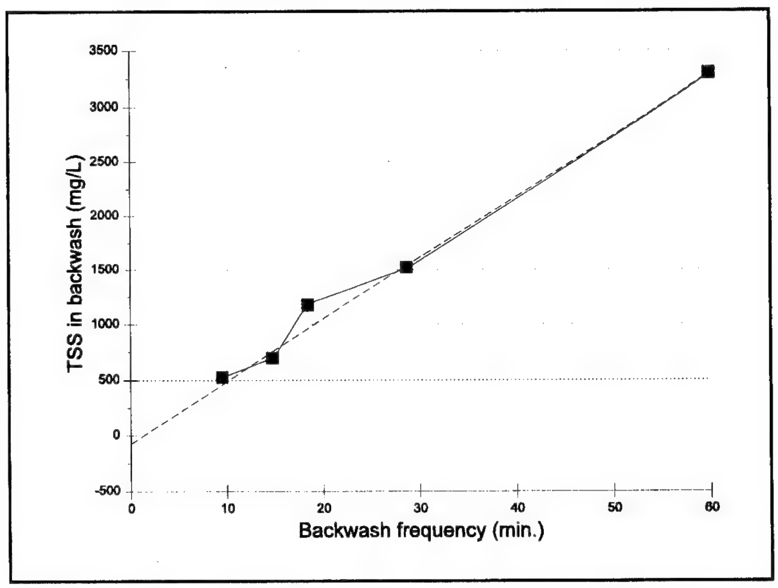

Figure 20. TSS vs. backwash frequency with constant filtrate flow (gal/min.).

Operation of the MF unit with effluent from the DeLaval centrifuges was successful and the optimum operational parameters were determined to be 182 L/m^2/hr (4 gpm) filtrate flux with a backwash frequency of 60 min. These operational parameters were established with a TMP of less than 5 psi, as recommended by Memtec. Filtrate quality exceeded expectations (0 to 10 mg/L TSS) while the maximum concentration of the NC in the backwash was less than expected (less than 0.5 percent NC fines in the backwash). However, in some of the tests, comparison of NC fines concentration in the

influent to the MF unit to the NC fines concentration in the backwash (concentrated effluent), indicates that greater than a 1000 fold concentration was achieved.

Site Evaluation

This effort involved processing DeLaval centrifuge influent (poacher pit effluent). At this location, the concentration of TSS was estimated to be 300 to 500 mg/L. A total of 11 tests were scheduled for the DeLaval centrifuge influent (tests 17 to 27). Filtrate flow rate from the unit was to be varied (1, 2, 2.5, 3, 4, 5 gpm) and backwash frequency was also to be varied (10, 15, 20, 30, 60 min intervals).

After numerous attempts, the poacher pit effluent could not be evaluated due to difficulties with NC particles plugging the 100 μm screen filter in the crossflow loop of the MF unit. A 100 μm bag filter was then installed in the feed line to the microfiltration unit to remove these large particles. It was concluded that sufficient quantities of large NC particles had apparently penetrated the bag filter since subsequent tests were also unsuccessful. In addition, the 100 μm bag filter was precoated with NC by passing the NC wastewater feed slurry through the filter for 2 days to increase the capability of the bagfilter to remove large NC particles. Subsequent attempts to run the MF equipment were also unsuccessful.

Several observations were made during processing of the poacher pit effluent. The wastewater could be processed without difficulty until the screen filter in the crossflow loop plugged with the large particles and restricted flow. Secondly, the filtrate fluxes of 1 to 5 gal/min could be processed with a TMP of 5 psi or less before the screen filter plugged. Plugging of the screen filter appeared to occur during the addition of poacher/blender house screening water.

Extended Operation Tests

This phase of the study involved operating the MF unit for an extended period of time under the optimum operating parameters established during site evaluation. These operating parameters were 4 gpm filtrate flow rate and 60-min backwash frequency for the DeLaval effluent. These conditions allowed an assessment of long-term capabilities to separate NC fines from wastewater. Additionally, it permitted a determination of the requirements for periodic caustic cleaning of the membranes to remove NC fines trapped in the pores or on the surfaces. Insufficient time was available to assess the service life of the membranes (estimated to be 1 to 2 years). (Memtech recommended caustic cleaning every 2 weeks based on its experience.)

Design Criteria Information and Economics Analysis

Design Criteria Information

Memtec was requested to develop design criteria information for a 1 million gal per day (MGD) MF facility for treatment of the DeLaval centrifuge effluent. The capabilities of the suggested MF plant were based on operational capacity and current use of the DeLaval centrifuges. Memtec provided the requested design criteria information and also estimated installation and operational costs for a treatment facility. Appendix C includes the Memtec design and estimated cost.

Memtec proposed a conservative design based on 50 percent of the maximum design capacity of the membranes (0.5 gpm/m^2) even though data developed during this study indicated that the system could be operated at 80 percent capacity (0.8 gpm/m^2). Memtec recommended that three 600 m^2 systems be installed. Each 600 m^2 block (type 600M10) would consist of 60 10m^2 modules. A Goulds 3171 or equal (submersible) pump would be necessary to provide the driving force to operate the system in both the forward and backwash modes. The proposed MF plant would include, in addition to the basic membrane/module block, appropriate manifolds for system connections, pumps, air compressors, software, process/control design criteria, overall design strategy, tanks, etc. All control panels and electrical components would be NEMA 4.

Economics

Memtec estimated that the specified MF facility would cost approximately $1,375,000. This value does not include installation costs and excludes building construction or preparation costs. The annual operating costs are estimated at $135,000 excluding maintenance and labor (which should be minimal). These costs are based on replacement of the modules every 3 years, cleaning chemicals (based on monthly cleaning), and electrical usage. Assuming that 5 million gallons per week (MGW) of DeLaval centrifuge effluent are processed, operating costs would be $0.52/1000 gal. Since the specified MF facility was designed to provide excess capacity, the treatment costs would drop to $0.26 /1000 gal, if the rate were increased to 10 MGW. No additional operator cost are anticipated since the MF unit could be operated concurrently with the DeLaval centrifuges.

6 Field Evaluation Using a Hollow Fiber Crossflow Microfiltration Unit With Prefiltration

The experiments outlined in Chapter 5 demonstrated that NC fines could be removed effectively from wastewater using a pilot Memtec Microfiltration (MF) unit. However, the technology was limited in application since large NC particles (> 100 micron) obstructed the filtrate flow through the MF membranes. These larger particles were present in the wastewater contained in the settling pits, but not in wastewater discharged from the DeLaval centrifuges. Using prefiltration technology, the potential exists for successful use of the MF equipment with all types of wastewater discharged from the NC purification operation. To take a more conservative approach, 50 micron was used as a cut-off limit in this study. (Research in this chapter was conducted by Hercules Aerospace Inc., RAAP).

Procedures

Selection of Prefiltration Technology

A total of 9 different separation techniques and associated equipment were identified and evaluated for feasibility: solid bowl decanter, screen bowl centrifuge, vacuum belt filter, conventional rotary vacuum filter (RVF), cartridge filter, hydroclone, large I.D. (lumen) MF, tilting pan filter, and Bird RVF. The decision matrix included equipment that could separate solids by using either filter media or centrifugal force. The criteria for evaluation included: safety, maintainability, overall cost, operational efficiency, particle size segregation, and applicability to NC.

A commercial facility was also visited to obtain additional information with a production scale Bird Machine Company Rotary Vacuum Filter (RVF). The plant uses a 100 sq ft RVF to reclaim product motive water. The NC-contaminated water is pumped to a holding tank. The contents of the tank are then fed to the RVF through a continuous flow loop. Effluent overflow from the RVF is returned to the holding tank for recirculation. The recovered NC is discharged from the RVF into another holding tank. The filtrate and air are drawn from the RVF into a vacuum separator. A blower

exhausts the air from the vacuum separator outside the building. The recovered liquid (filtrate) is pumped from the vacuum separator back into the manufacturing process.

Using the above matrix, Hercules, Inc. selected two preferred filtration technologies for evaluation: the Bird RVF and the Ronnington-Petter* cartridge type filter unit. Economics, safety, technical feasibility, and a hazards analysis were completed to make a qualitative comparison of the two technologies considering MF vendor's recommendation. A safety comparison showed that either filter could be installed and operated to meet Army and Hercules safety criteria.

Based on the literature review and manufacturer's recommendations, it was concluded that laboratory scale "leaf tests" would be needed to evaluate the performance of each separation technology.

Bird Machine Company RVF

The RVF used during the pilot-scale experiments consisted of a variable speed rotating drum covered with replaceable filter media. The filter drum is evacuated by a liquid sealing ring vacuum pump to an approximate vacuum level determined by the filtration capacity of the equipment. The filter drum rotates, subjecting the filter media to a continuous cycle of solids separation, solids drying, and solids discharge.

A cake of solids forms on the filter media while filtrate is drawn into the filter drum and vacuum separator. The filter drum continues to rotate, removing the filter media from the slurry, drawing air through the solids cake, and lowering moisture content. The solids cake is pneumatically discharged, thus renewing the filter media. The filtrate and air are separated in the vacuum separator. Filtrate is removed by a centrifugal pump and the air is removed by the liquid sealing ring vacuum pump.

Qualification of Filter Media

The RVF was operated in conjunction with the Memtec MF unit during all experiments (Figure 16). Previous experience using the Memtec unit indicated that larger NC fibers would reduce the filtration capability of the MF equipment. As previously discussed, to operate the MF unit at full capacity, it was necessary to use a prefilter such as the RVF to remove the larger particles from the wastewater flowstream. By varying the type of filtration media used on the RVF, it was possible to separate different quantities of solids from the wastewater flowstream.

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* Ronnington-Petter Inc., a Division of Dover Corp., P.O. Box 18-T, Portage, MI 49081, FAX 616/323-2403, tel. 616/323-1313.

Using leaf test results, Tetko Inc. No. 7-76-SK22 and No. 7-21/15 filter medias were selected as primary and secondary choices for installation and evaluation on the rotary vacuum filtration equipment.

Equipment Installation

A pilot-scale RVF with a 1 sq ft wetted surface area was rented from Bird Machine Company and used during these experiments. This pilot-scale unit was skid-mounted and required a source of 440-volt, 3-phase electrical power for operation. The skid mounted unit and control panel were installed in the basement of Building 3024 (Figure 21). This building contains both poaching and blending operations. The following effluents were accessible from this building: Jordan beater decant effluent, poacher decant effluent, and wringer effluent.

Process Flow Diagrams

To obtain Jordan beater decant water in the building where the RVF was installed, it was necessary to pump NC slurry from the Jordan operation to the poacher/blender building without any previous decanting operation. The Jordan decant water was then

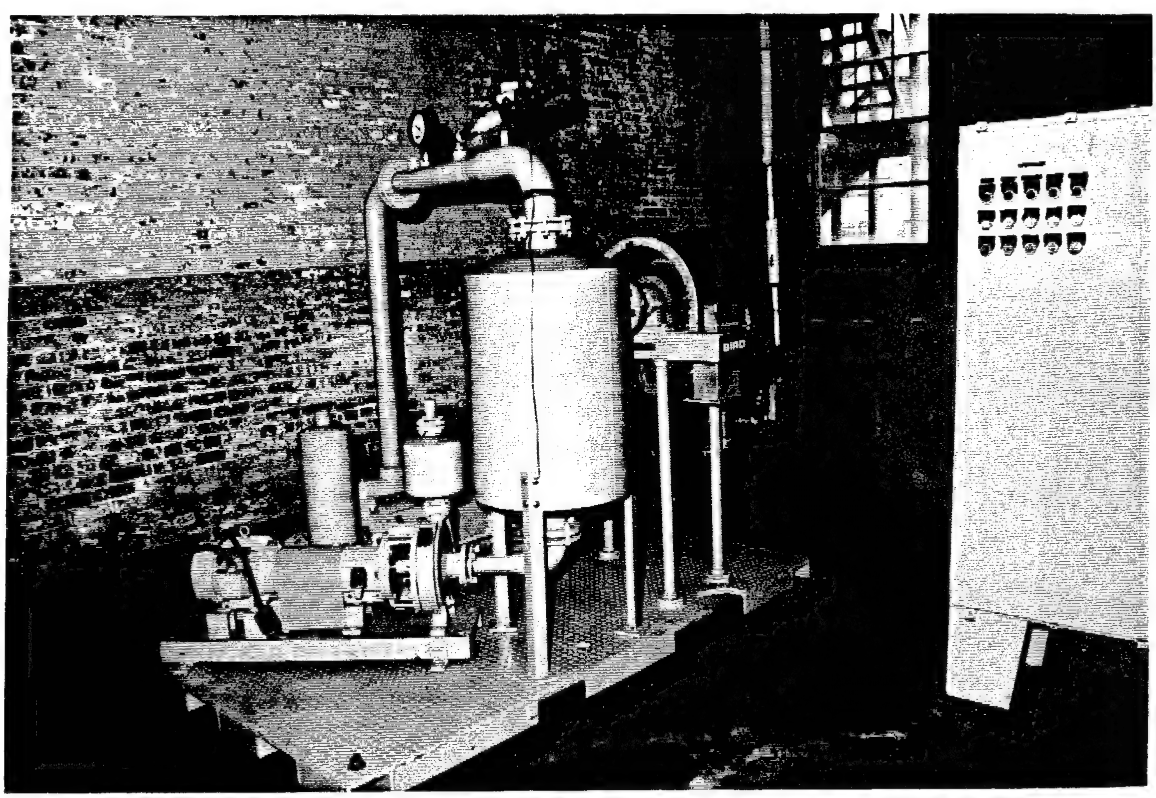

Figure 21. Pumps and tanks associated with the Bird rotary vacuum filter.

siphoned from a poacher tub to the RVF and MF unit before any poacher treatment began. Figure 22 shows a process flow diagram for the operation.

The wastewater samples, which included influent to the RVF, effluent from the RVF, and effluent from the MF unit, were tested for TSS, chemical oxygen demand (COD), and pH. NC solids from the RVF discharge were tested for fineness, freeness, nitrogen content, and heat stability.

Test Plan

A test plan consisting of four different phases: water, poacher effluent, Jordan beater effluent, and final wringer effluent was developed to evaluate the RVF and MF equipment. The test matrix maximized operating time with each type of effluent.

The goals of the test plan for the RVF and MF equipment were to:

1. Determine the amount of NC in wastewater effluent at each process location (Jordan cutter, poacher, final wringer) before treatment with the RVF.
2. Concentrate the NC from the individual flowstreams using the equipment and determine the reuse potential.
3. Determine the process water reuse potential. Recycled water must not cross-contaminate the various grades of NC. Evaluation of pH and TSS will determine the reuse potential of process water.
4. Determine optimum operating conditions with effluent from the locations stated in item number 1 (above) for NC from cellulose linters and wood pulp.

Results and Analysis

Phase 1, Water Tests

The water tests consisted of two evaluations performed simultaneously on both the rotary RVF and MF units prior to operations using wastewater effluent containing NC. The RVF was connected to a supply of nonpotable water during the tests. Tetko, 40 micron, PRD woven cloth was installed on the filter drum. The filtrate from the RVF was continuously pumped from building 3024 to the MF unit in building 3025.

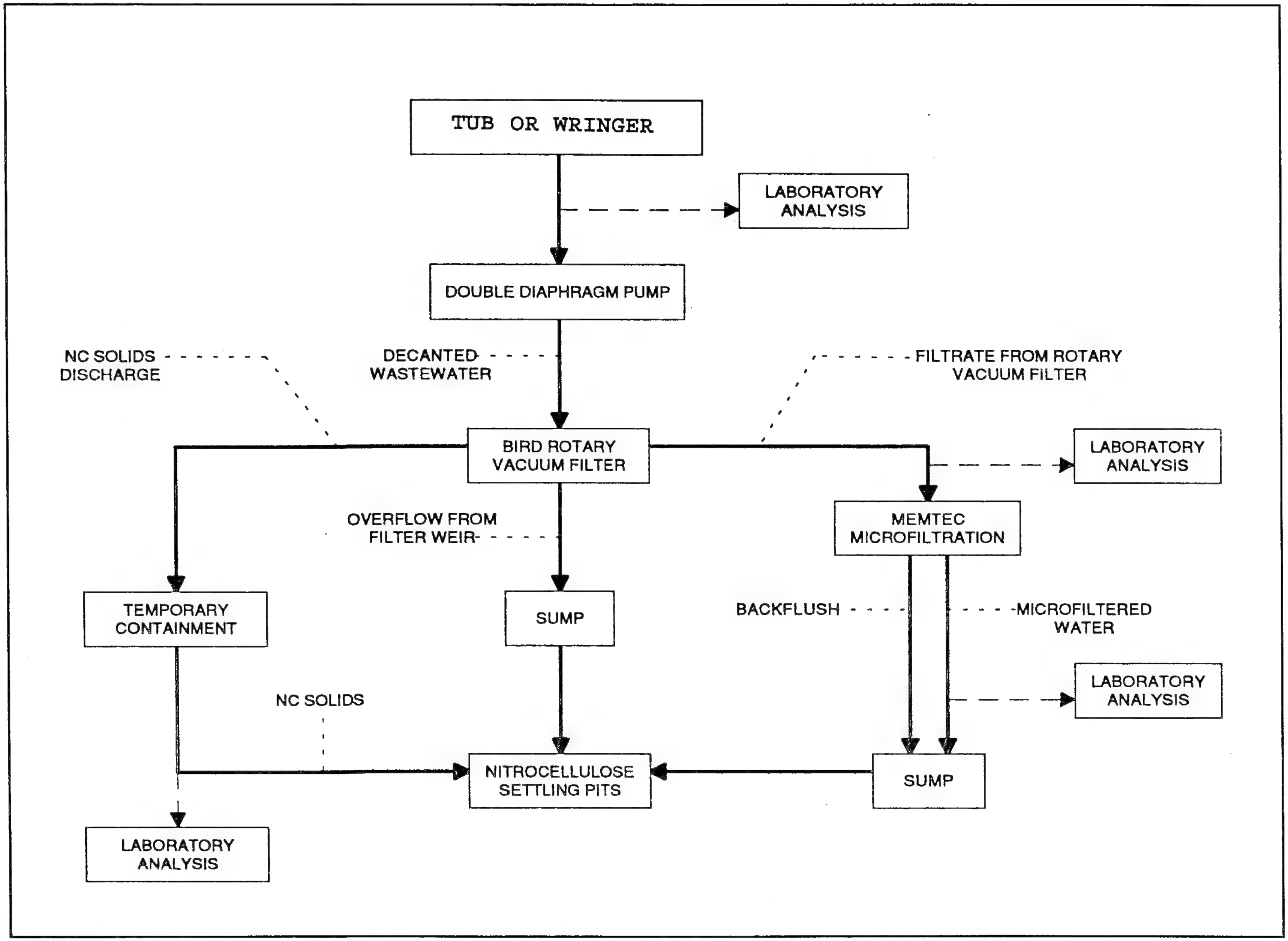

Figure 22. Process flow diagram of the rotary vacuum filter and microfiltration equipment with poacher and jordan cutter decant wastewater.

Phase 2, Poacher Decant Effluent Tests

Pulp-Based NC. The first wastewater effluent processed simultaneously by both the RVF and MF unit was from a poacher tub containing P1, a pulp-based NC. The RVF drum was dressed with Tetko Inc. No. 7-76-SK22, with a mean filtration rating of 29-31 microns. Tests number 3 through 8 varied operating time (120 and 240 min), sample frequency (60 and 120 min), and filtrate flowrate (8 to 20 gpm) for the RVF. For the MF equipment, operating time, sample frequency, and filtrate flowrate (3 to 5 gpm) were varied.

The effluent in the poacher tub was approximately 170 °F during tests 3, 4, and 5. The high temperature of the effluent was due to the fact that the tub was drained hot after completion of individual boils. This temperature exceeded the maximum operating temperature of the MF unit and caused frequent shutdowns. The hot effluent had no effect on operation of the RVF. To counter the effects of the hot effluent, the flowrate to the RVF was limited to 8 gpm. This allowed the effluent to cool while being pumped to the MF equipment. In normal production operation, the poacher effluent is decanted to the poacher settling pits at an elevated temperature, without any anomalous effects.

The poacher tub was allowed to cool before tests 6, 7, and 8. In normal production operation, the NC in the poacher tub is allowed to settle a minimum of 2 hours before beginning decanting operations. To counter the effects of the increased settling time while the tub was cooling, the tub contents were agitated for several minutes then allowed to settle for 2 hours prior to operating the RVF and MF equipment. After the poacher tub was allowed to cool, the flowrate of the effluent pumped to the RVF was increased to 20 gpm. With a backflushing rate of once every 7 minutes, the maximum observed TMP in the MF unit was less than 2 psi.

A total of 36 wastewater samples, which included influent to the RVF, effluent from the RVF, and effluent from the MF unit, were tested for TSS, COD, and pH. The pH in RVF influent, RVF effluent, and MF effluent varied from 6.1 to 7.8, from 6.5 to 7.6, and from 6.7 to 7.8, respectively. Although the COD and the TSS varied in MF influent (RVF effluent) (350 to 600 mg/L COD and 300 to 590 mg/L TSS), MF constantly produced an effluent with about 220 mg/L COD (Figure 23) and zero mg/L TSS (Figure 24). Figure 24 also shows that only a small percentage of TSS was removed by RVF. However, such an activity did ensure the normal performance of MF.

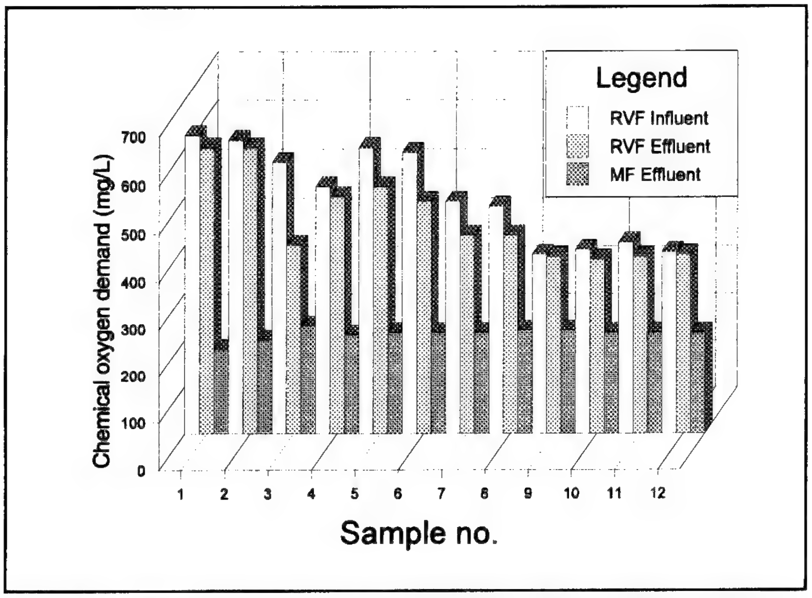

Figure 23. Filtration of poacher tub decant effluent–COD (P1 type NC).

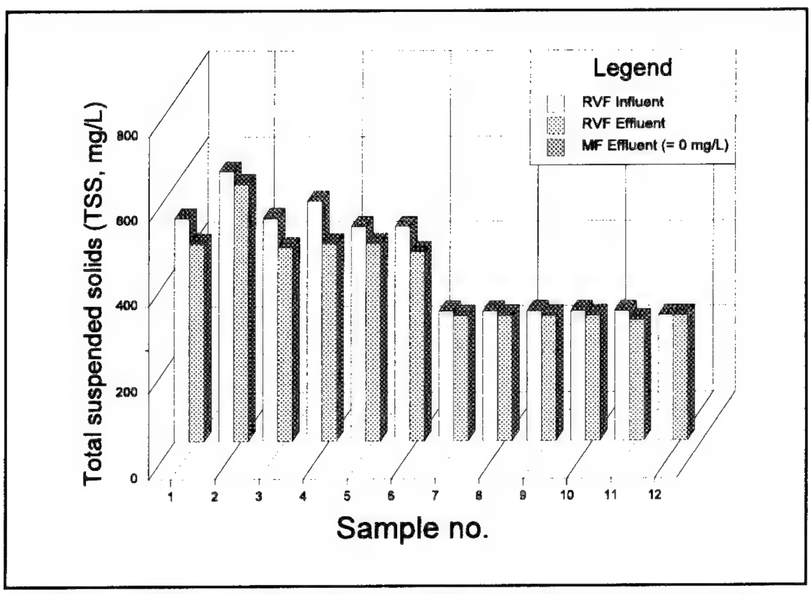

Figure 24. Filtration of poacher tub decant effluent–TSS (P1 type NC).

TSS for 12 samples ranged from 372 to 568 ppm and vacuum was maintained at 3 in. Hg. An increase in the filter drum vacuum would indicate that a substantial solids cake or other obstruction had reduced flow through the filter drum. Two variables may be changed to maintain a constant vacuum level in the filter drum; vary the flowrate to the filter weir and/or vary the rotational speed of the filter drum. In all experiments, the rotational speed of the filter drum was held constant and the influent flowrate to the RVF was varied by small amounts during a specific test to maintain a relatively constant vacuum of 3 in. Hg. in the filter drum.

During tests using the P1 type NC poacher effluent, the RVF operated far below the maximum filtration capacity with the filtration media used and the fixed 3 rpm drum rotational speed. However, the MF unit performed poorly when the temperature of the filtrate from the RVF exceeded 120 °F.

Lint-Based NC. The second effluent processed through the RVF and MF units was from a poacher tub containing BL7 NC, manufactured from cotton linters. The effluent was allowed to cool before equipment operation, i.e., the poacher tub contents were agitated and allowed to settle for 2 hours prior to operations. Tests 9 and 10 were conducted concurrently using Tetko No. 7-76-SK22 as the filter media. At the conclusion of test No. 10, a visible amount of NC was retained on the RVF filter media. Efforts to dislodge these solids using the pneumatic discharge were unsuccessful, indicating the filter media had become "blinded." Long-term operation using this filter media would be impractical. As a result, this filter media was replaced with Tetko No. 7-21/15.

Tests 11 through 14 were conducted with the No. 7-21/15 filter media. Tests number 9 through 14 varied operating time (120 and 240 min), sample frequency (60 and 120 min), and filtrate flowrate (4 to 15 gpm) for the RVF. These tests also varied operating time, sample frequency, and filtrate flowrate (3 to 5 gpm) for the MF equipment. The frequency of MF backflushing was reduced to once every 15 minutes.

The COD in MF influent varied from 150 to 490 mg/L, and the COD in MF effluent varied from 5 to 90 mg/L (Figure 25). The TSS in MF influent varied from 150 to 500 mg/L, and the TSS in MF effluent was consistently constant at 0 mg/L TSS (Figure 26).

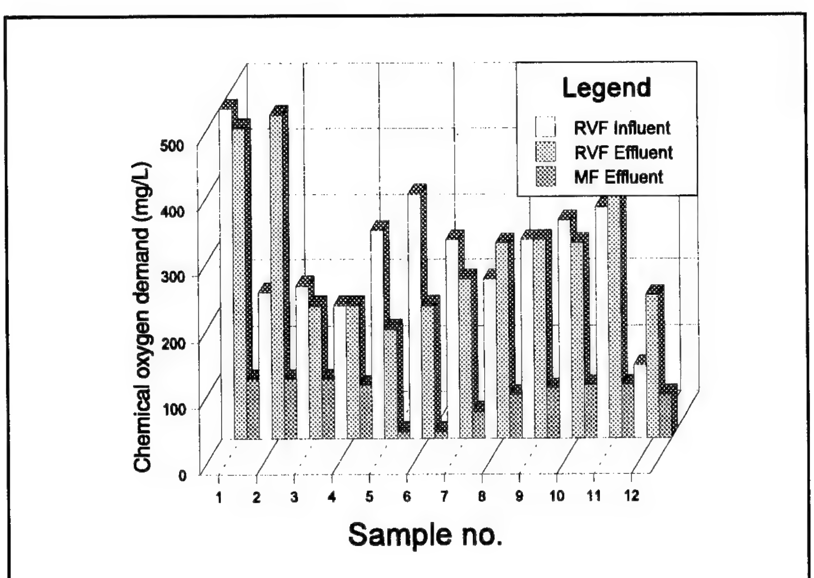

Figure 25. Filtration of poacher tub decant effluent–COD (BL7 type NC).

Figure 26 shows that the TSS removal by RVF varied significantly, likely due to the variation in the particle size distribution in the influent to RVF.

NC solids from tests 9 through 14 were combined before analysis. The test results for BL7 type NC recovered from the RVF discharge are compared to the test results for the BL7 type NC remaining in the poacher tub. In each case, the NC recovered from the RVF meets applicable acceptance requirements for this NC type.

Figure 27 shows the relationship between the TSS in the flowstream to the RVF and any corresponding increase in filter drum vacuum. During experiments using the wastewater contaminated with BL7 type NC, a distinct increase in the filter drum vacuum occurred when the influent wastewater contained solids above 290 ppm. This indicated that a substantial solids cake or other obstruction had reduced flow through the filter drum. Under this set of conditions, the filtering capacity of the RVF was exceeded when the influent contained 409 ppm or more of solids. Therefore, the RVF is incapable of filtering over 15 gpm of BL7 NC-contaminated poacher wastewater if a Tetko No. 7-21/15 filter cloth is used and the filter drum rotational speed is restricted to 3 rpm. The MF unit easily operated at the rated design capacity of 5 gpm (227/m^2/hr) during tests 10, 11, 12 and 14.

Phase 3, Jordan Beater Decant Effluent Tests

The pulp-based NC (P1 type) and lint-based NC (BL1 type) wastewater were pumped from the Jordan beater operation in building 1022 to a tank in Building 3024 prior to filtration operations. The RVF drum was dressed with Tetko Inc. No. 7-21/15 filter fabric during testing. Tests number 15 through 26 varied operating time (120 and 240

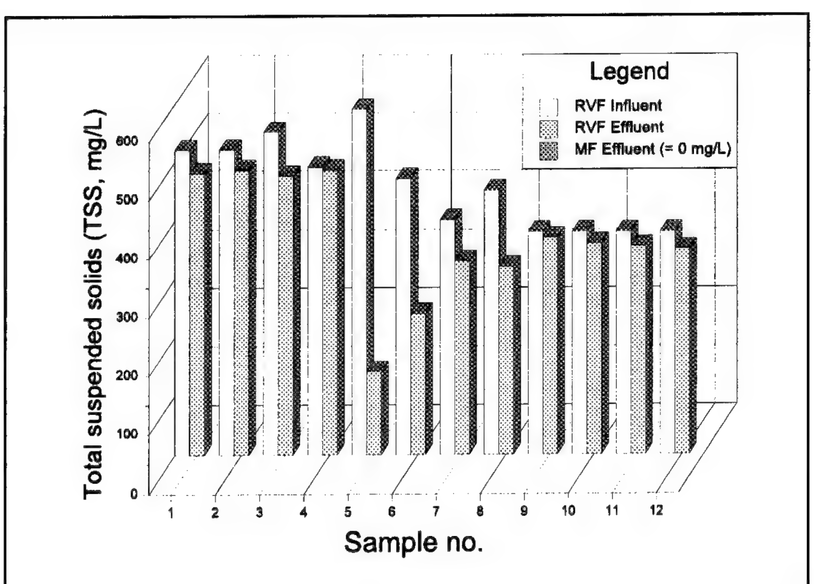

Figure 26. Filtration of poacher tub decant effluent–TSS (BL7 type NC).

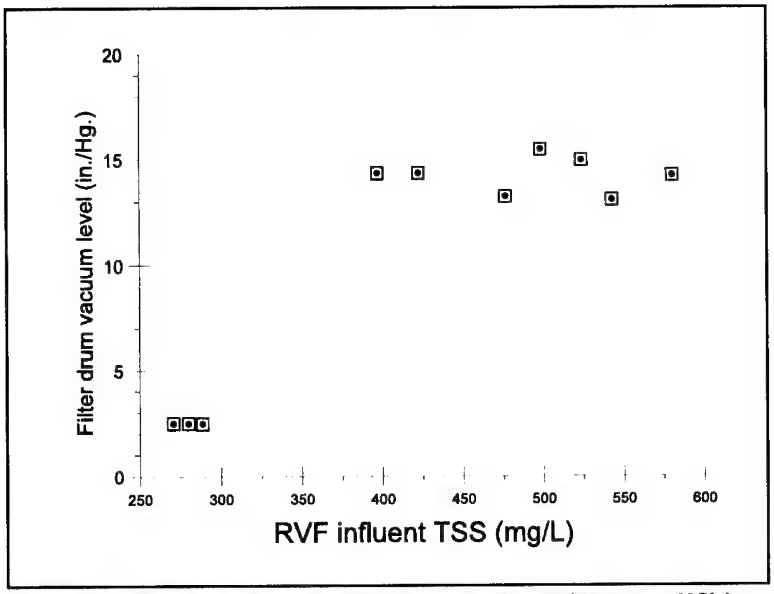

Figure 27. Filtration of poacher tub decant effluent (BL7 type NC) by RVF: TSS vs. filter drum vacuum level.

min), sample frequency (60 and 120 min), and filtrate flowrate (10 to 20 gpm) for the RVF. These tests also varied operating time, filtrate flowrate, and sample frequency for the MF equipment. The frequency of MF backflushing was once every 15 minutes.

Phase 4, Final Wringer Effluent Tests

The effluent used during operations with P1/P7 and BL4 type NC was obtained by tapping a wringer wastewater line in Building 3026. Under the conditions experienced during these tests, the RVF was able to remove a large percentage of the NC particles from the wastewater while operating below maximum design capacity. The filtrate from the RVF was relatively clear, and the MF unit was not required to remove many solids from the effluent. Therefore, the MF equipment easily operated at the rated design capacity of 5 gpm (227 $L/m^2/hr$) during tests 27 through 38.

Concept Design Study

Design Criteria Information

At several of the process buildings, three types of NC are processed concurrently during normal production conditions. Therefore, three independent systems would be required at these locations. However, to maintain production flexibility and interchangeability of parts during maintenance, it is recommended that all three systems be sized the same.

General specifications for Bird Machine Company rotary vacuum filters are based on final design parameters required on similar machines used for processing potentially explosive materials. These specifications are:

1. Material of construction: 304L stainless steel for all wetted parts
2. Hood access door and chain guard: nonsparking aluminum
3. Special open compartment center girt for wash out
4. Stainless steel hood with aluminum quick-access door with clamps
5. Two wash-down headers
6. Drive mounted on top of bearing stands
7. Special sealed water compartment between head trunion and girt
8. Special "cotton ball" finish and ground and polished welds on internal parts
9. All surfaces inside and out:
   a. Safety wired fasteners
   b. Plugged valves for smooth surfaces
   c. Machine ground
   d. Explosion proof drive, and AC variable frequency controller with NEMA 7/9 remote station.

Concepts

Four basic concepts for regular peacetime production, concepts A through D, emerged from the concept design study using MF and RVF. Concept A uses all of the NC extracted (fibers and fines) according to type and blend (blend specific) so that it can be placed back into the product rather than becoming pit cotton. Concept B uses only the large fibers that are removed with RVF for recycling back into the product. The small fines that never settle are discharged to the poacher pit and treated for disposal.

Concept A represents blend-specific MF without poacher water recycling. This concept is based on the precept that all of the NC (fiber and fines) in the effluent streams that normally go to the poacher settling pits could be recovered and placed back into the product. This process must be performed without cross-contaminating different types of NC being produced. Figure 28 shows a flow diagram of concept A for wood pulp. A similar diagram can also be drawn for cotton linters.

Concept A is also based on the precept that all effluent water, except the water from the boiling tub and poacher houses, should be recycled. Since the boiling tub process water is acidic and causes the NC to quickly settle out, the effluent does not contain as much suspended solids as in subsequent operations. Any solids that go into the boiling tub settling pit are easily collected. Therefore, evaluation of the RVF and MF units with boiling tub effluent was not a part of the project's scope of work.

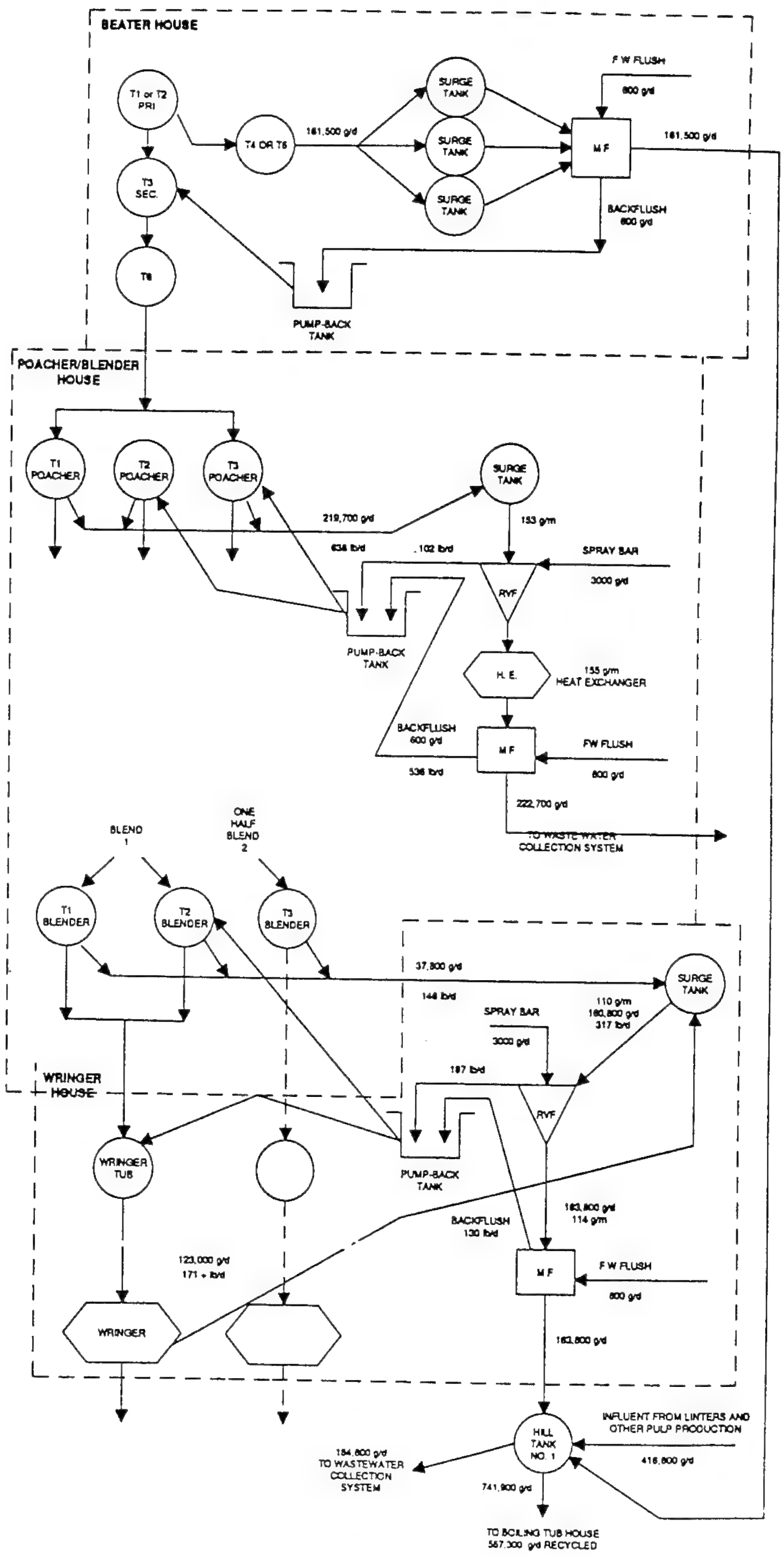

Figure 28. Concept A flow diagram for pulp NC blend-specific rotary vacuum filtration and blend-specific microfiltration without poacher water recycling.

Since the effluent wastewaters from the boiling and poaching tubs are primarily wash waters, these effluents are not considered viable candidates for recycling. The risk of affecting product quality is assumed to be too great to justify the cost savings of only $0.43 per 1000 gal of water (1992 rate) that could be realized.

Concept A would allow the water recovery system installed in the 1970s to be used as designed. The system, which consists of the DeLaval centrifuges, collection pits, hill tanks, piping to the boiling tub house, and distribution system to the individual tubs, was not used because of cross-contamination of products. The DeLaval centrifuges have proven to be very effective in production. However, the effluent from the centrifuges contains approximately 30 mg/L of fibers and fines, which is unacceptable.

Concept A allows the RVF and MF units to replace the DeLaval centrifuges, so the rest of the water recovery system could be used to recycle the filtered water. The poacher pit may possibly be used as a repository for the filtered water. The same pumping system used to pump the water from the DeLaval centrifuges to the hill tanks could be used to pump the filtered water from the pit to the hill tanks instead. The piping system from the hill tanks to the boiling tub houses and the internal distribution system in the boiling tub houses may be used with only slight modifications.

According to calculations using the water balance data and production records, approximately 510,000 gal/day of filtered water was used in 1992 for motive water (fire hoses) at the boiling tub house. Approximately 47,000 gal/day of additional water was used for tub/line washes for a total of 557,000 gal/day. According to the flow diagram for concept A, approximately 742,000 gal/day could be recycled leaving an excess of 185,000 gal/day. The excess would be equivalent to a 25 percent blowdown.

An alternative would be to use the recycled water at other locations, in addition to the boiling tub house, for motive water and tub/line washes. This alternative would require a more complex distribution system. In this scenario, however, approximately 214,000 gal of water would be needed in addition to the 510,000 gal/day of motive water for a total of 724,000 gal/day. An excess of approximately 18,000 gal/day would be equivalent to only a 2.4 percent blowdown. The amount of blowdown could be increased simply by using fresh water instead of recycled water for tub/line washes at one or more of the processing buildings.

At the poacher house, the maximum number of different types of NC that are processed concurrently during normal production conditions is three. Therefore, three independent systems consisting of a surge tank, RVF unit, heat exchanger, MF unit, and pump-back tank (Figure 28), would be required to treat the decant water prior to being discharged to the wastewater collection system.

The filter cake of NC fibers periodically removed from the filter cloth of the RVF, located on the ground floor of the building, would be collected in a plastic tote tub. Several times a shift, the plastic tub would be placed onto a small vertical reciprocating conveyor (VRC) or freight elevator and sent to the second floor. On the second floor, the contents of the tub would be emptied back into the appropriate poaching tub by an operator. An alternate method would be to discharge the NC directly into an agitated pump-back tank along with the fines from the MF unit as described below.

The effluent from the RVF would then be pumped through a heat exchanger to reduce the temperature of the water from approximately 180 °F to 120 °F, or less. Due to temperature restrictions on the potting compound used in the Memtec MF unit, a temperature limit switch is installed in the MF controls that shuts the unit down when the maximum temperature is reached. The backflush from the MF unit, containing the filtered NC fines, would be plumbed to a pump-back tank that would pump the water to a flexible hose on the second floor. The hose would be placed into the appropriate poacher tub by an operator.

Since a 60 °F reduction in the temperature of water flowing at a rate of approximately 120 to 150 gal per minute results in significant heat extraction, methods of using this heat should be considered in the final system design. Preheating the water used to wash the NC between boils at the poacher house is one potential use.

In 1984, a heat transfer system was designed, installed, and evaluated in the NC purification process at RAAP (Johnson and Ogle, 1984). The system, consisting of a four-unit heat pipe heat exchanger (HPHX), was used to transfer the heat from the hot wastewater from the boiling tub house to the incoming wash water. The system performed in accordance with theoretical design parameters. Under optimum conditions, a heat transfer rate of 7,700,000 Btu/h was attained saving approximately 27 percent of the steam required to bring the contents of a boiling tub to boiling.

The HPHX system was never used in production due to problems with NC fibers and fines plugging the lines. However, rotary vacuum filtration of the water before it enters the HPHX unit, as in concept A, may eliminate this problem and make the system viable for consideration in the final design for the poacher house.

At the wringer house, three types of NC can be processed at a time under normal production conditions. Therefore, three systems consisting of a surge tank, RVF, MF, and a pump-back tank would be required. Figure 28 shows how each system must also handle the water normally decanted at the blender house. According to procedure, the blender tubs are agitated for 6 hours, and then the NC is allowed to settle.

Approximately 3 to 4 ft of water is decanted off to thicken the mixture before transferring it to the wringer house. This decant water would have to be pumped directly to a surge tank in the wringer house instead of to the poacher pits. Any additional water decanted at the wringer tub, and the water removed from the NC at the wringer would also be pumped to the surge tank for filtering before being pumped to the hill tank(s).

Concept B assumes that the small fines that pass through the filter cloth of the RVF units would create problems either by affecting product quality, or by not being adequately retained at the various pump-back locations or at the dehydration pressing operation. If the fines are not eventually bound up in the product, they may enter the dehy solvent recovery system where they can plug the alcohol recovery lines or accumulate excessively in the spent alcohol distillation tower.

Alternative Concept for Maximum Production

Under maximum production conditions, eight different types of NC could be in process at the poacher/blender house. To prevent cross-contamination, eight separate filtration systems would have to be installed. Each system would consist of a surge tank, RVF unit, MF unit, pump-back tank, and ancillary equipment. Ancillary equipment would include such things as vacuum pumps, filtrate pumps, and drive motors. Installation of this amount of equipment would require a large auxiliary building for each line.

The fibers and fines from wood pulp types of NC, such as P-1 and P-7, could be segregated as unadulterated pit pulp for use in any of the several single-base propellants such as M-1 and M14. Specifications for these propellants already allow the use of pit cotton containing cotton linters and wood pulp.

The fibers and fines from cotton linters production could be segregated as an unadulterated pit lint (no pulp) for use in any of the several types of multibase propellants that require blends of various types of cotton linters. For example, the specification for M7 propellant, which requires a blend of BL1 and BL7, could potentially be revised to allow unadulterated pit cotton to be blended into the mixture without significantly affecting the performance characteristics of the propellant. Specifications for these propellants do not currently allow the use of pit NC; requalification of these items may be required.

7 Hazards Analysis

Prefiltration Process

The Hazards Analysis Department of Hercules Aeorspace, Inc. performed a safety comparison of the Bird RVF and Ronnington-Petter multiplex filter unit. The overall conclusion of this qualitative evaluation is that the Ronnington-Petter filter could be more easily installed and operated to meet applicable safety criteria than the Bird RVF. Both units would be acceptably safe provided they were properly modified, installed, and operated.

Microfiltration Process

Hazards Analysis also performed a Preliminary Hazards Analysis (PHA) and a quantitative Risk Analysis of the Memtec crossflow microfiltration unit. The conclusions were: (1) During normal operations, the NC fines are suspended in water (>99 percent water-wet), which will prevent sustained burning or propagation of a burning reaction, (2) The pilot-scale microfiltration equipment and its operation are acceptably safe, and (3) Potential hazards resulting in personnel injury or equipment damage would involve abnormal events that could cause accumulation, drying, and initiation of NC in or outside of the equipment.

8 Summary and Conclusions

Application of crossflow microfiltration (MF) technology for the separation of NC fines from wastewater was investigated using bench-scale flat-sheet MF units and a pilot-scale hollow fiber Memtec MF unit. Overall, it was concluded that NC fines could be separated effectively by MF. The technology, however, was susceptible to large NC particles, and prefiltration was needed.

Specific conclusions drawn from the bench-scale study using the flat-sheet MF units include:

1. A high crossflow velocity resulted in the reduction of cake layer formation. However, it was not sufficient to deter the membranes from performance deterioration. The crossflow velocity of about 1.07 m/sec is recommended for this situation.
2. A higher transmembrane pressure (TMP) drop could lead to increased membrane fouling resistance for the flat-sheet MF unit.
3. MF membrane pores were susceptible to blockage by NC fines due to the wide range of NC fine particle size distribution.
4. The larger pore size membrane (0.65 μm) performed better than 0.2 μm and 0.45 μm pore diameter membranes, while all the membranes produced effluents with almost similar turbidity.
5. It was possible to concentrate NC fines to a great extent; however, increased turbidity in the feedwater created higher membrane fouling rate, suggesting a potential need for frequent cleaning.
6. An increase of the permeate flux was observed when the pulsating cleaning was applied with an on/off running cycle and interval. From this, it is concluded that a long-term practical permeate flux level could be maintained by incorporating a pulsating cleaning technique combined with chemical cleaning.
7. A model combining the concepts of diffusivity and the cake filtration theory was proposed to explain the experimental observations and to understand the physical phenomena governing the permeate flux. Further study is necessary to quantitatively predict the permeate flux using the equation proposed in this study.

The specific conclusions that were drawn from the pilot-scale studies using the Memtec MF unit (polypropylene hollow tubes with 0.2 μm pores) include:

1. Operation of the MF units with effluent from the DeLaval centrifuges was successful. The optimum operational parameters were determined to be 4 gpm (182 L/m^2/hr) with a backwash frequency of 60 min. These operational parameters were established with a TMP of less than 5 psi, a filtrate quality of 0 to 10 mg/L TSS, and a maximum concentration of the NC in the backwash of about 0.34 percent.
2. Operation of the MF unit with poacher pit effluent (DeLaval centrifuges' influent) was unsuccessful because the screen filter in the crossflow loop was plugged with the large NC particles. This led to the subsequent study on MF with prefiltration for separation of NC fines from the wastewater streams containing these particles.
3. The Bird Company Rotary Vacuum Filter (RVF) was selected as a desirable prefiltration option for MF. The NC contained in the wastewater streams from the poacher and wringer operations were efficiently concentrated using the RVF equipment.
4. The MF equipment operated as specified by the vendor and was relatively reliable. The MF unit shut down when fed poacher effluent in excess of 120 °F. The MF modules were successfully regenerated by backflushing.
5. The hazards analysis performed on the RVF and the MF showed that both units would be acceptably safe provided they were properly modified, installed, and operated.

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Appendix A: Manufacturers of Microfiltration Membranes and Systems

Manufacturer	Address	Phone #
A/G Technology Corp.	101-T Hampton Avenue Needham, MA 02414	(617) 449-5774 (800) 248-2535
Amicon Corp.	72 Cherry Hill Drive Beverly, MA 01915	(508) 777-3622 (800) 343-0696
CeraMem	12 Clematis Avenue Waltham, MA 02154	(617) 899-0467
Consler Corp.	306 West Main Street Honeoye Falls New York, NY 14472-0351	(800) 321-4789 or (800) 852-1379 (in NY State)
Cuno Microfiltration Products	400 Research Pky. Meriden, CT 06450	(203) 237-5541
Domnick Hunter Inc.	6636-D East W.T. Harris Blvd. Charlotte, NC 28215	(704) 568-8788 (800) 345-8462
Gelman Sciences	600 S. Wanger Road Ann Arbor, MI 48103	(313) 665-0651 (800) 521-1520
LSL Biolafitte	8500 Evergreen Blvd. Minneapolis, MN 55433	(612) 786-0302
Membrex Inc.	155 Route 46 West Fairfield, NJ 07004	(201) 575-8388
Memtec America (MEMCOR)	9690 Deereco Road, Suite 700 Timonium, MD 21093	(410) 560-3000
Mciro-Filtration Inc.	401 McCormick Dr. P.O. Drawer 615 Lapeer, MI 48446	(810) 667-3600
Microgon, Inc.	23152 Verdugo Drive Laguna Hills, CA 92653	(714) 581-3880
Millipore Corporation	80 Ashby Road E2DS Bedford, MA 01730	(617) 275-9200 (800) 645-5476
MIS (Micron Separation Inc.)	135 Flanders Road L P.O. Box 1046 Westborough, MA 01581	(800) 444-8212

New Brunswick Scientific Co.	Box 4005, 44 Talmadge Road Edison, NJ 08818	(800) 631-5417
NGK - Locke, Inc.	1000 Town Center, Suite 1650 Southfield, MI 4875	(313) 325-7210
Norton Performance Plasitc Co.	150 Dey Road Wayne, NJ 07470	(201) 696-4700
Nuclepore Corp.	One Alewife Center Cambridge, MA 02140	(800) 882-7711
Osmonics	7120 Henry Clay Blvd. Liverpool, NY 13088	(315) 451-0592
Osmonics	5951 Clearwater Dr. Minnetonka, MN 55343	(612) 933-2277 (800) 351-9008
Pall Corporation	2200 Northern Blvd. Easthills, NY 11542	(516) 671-4000
Poretics Corp.	111 A Lindbergh Ave. Livermore, CA 94550	(800) 922-6090
Porex Technologies Corp.	500 Bohannon Road Fairburn, GA 30213	(404) 964-1421
PTI Technologies	2323 Teller Road P.O. Box 2000 Newbury Park, CA 91320	(805) 499-2661
Rochem	3904 Del Amo Blvd., Suite 801 Torrance, CA 90503	(310) 370-3160
Sartorius Corp.	131 Hardland Edgewood, NY 11717	(800) 227-2842
Schleicher & Schuell	10 Optical Ave. Keene, NH 03431	(603) 352-3810 (800) 245-4024
Wheelabrator Engineered System Inc.	28 Cook Street Billerica, MA 01821	(508) 667-2828
W.L. Gore & Associates	P.O. Box 1550 Elkton, MD 21921	(410) 392-4440
U.S. filter	181 Thorn Hill Road Warrendale, PA 15086	(412) 772-0086
Zenon Environmental System Inc.	845 Harrington Ct. Burlington, ON, CA L7N 3P3 (NR)	(905) 639-6320 (800) 265-6369

Appendix B: Test Matrix and Experimental Results for Removal of NC Fines From the Influent and Effluent of Delaval Centrifuges by Crossflow Microfiltration at RAAP, Radford, VA

Table B1. Test matrix for crossflow microfiltration of NC fines.

Test No.	Test influent	Estimated NC conc. (mg TSS/L)	Run Period (min)	Sample frequency (min)	Total No. samples (inf. and eff.)	Filtrate flow rate (gal./min)	Process volume (gal.)	Backflush interval (min)	Sample analysis	freq/backwash	
										System press.	Flux
Phase I - Water											
1	water	0	15	0	0	2.5	15	15	-	2	2
2	water	0	15	0	0	5	75	15	-	2	2
3	water	0	30	0	0	10	30	15	-	2	2
4	water	0	30	0	0	5	150	15	-	2	2
5	water	0	60	0	0	5	300	15	-	2	2
Phase IIA - Site Evaluation											
6	cent. eff.	80	240	120	4	1	240	15	TSS	2	2
7	cent. eff	80	240	120	4	2	480	15	TSS	2	2
8	cent. eff	80	240	120	4	2.5	600	15	TSS	2	2
9	cent. eff.	80	240	120	4	3	720	15	TSS	2	2
10	cent. eff.	80	240	120	4	4	960	15	TSS	2	2
11	cent. eff.	80	240	120	4	5	1200	15	TSS	2	2
12	cent. eff.	80	240	120	4	3*	720	10	TSS	2	2
13	cent. eff.	80	240	120	4	3*	720	15	TSS	2	2
14	cent. eff.	80	240	120	4	3*	720	20	TSS	3	3
15	cent. eff.	80	240	120	4	3*	720	30	TSS	4	4
16	cent. eff.	80	240	120	4	3*	720	60	TSS	3	3
Phase IIB - Site Evaluation											
17	poacher pit eff.	300	240	120	4	1	240	15	TSS	2	2

Table B1. (Cont'd).

Test No.	Test influent	Estimated NC conc. (mg TSS/L)	Run Period (min)	Sample frequency (min)	Total No. samples (inf. and eff.)	Filtrate flow rate (gal./min)	Process volume (gal.)	Backflush interval (min)	Sample analysis	freq/backwash System press.	freq/backwash Flux
18	poacher pit eff.	300	240	120	4	2	480	15	TSS	2	2
19	poacher pit eff.	300	240	120	4	2.5	600	15	TSS	2	2
20	poacher pit eff.	300	240	120	4	3	720	15	TSS	2	2
21	poacher pit eff.	300	240	120	4	4	960	15	TSS	2	2
22	poacher pit eff.	300	240	120	4	5	1200	15	TSS	2	2
23	poacher pit eff.	300	240	120	4	3*	720	10	TSS	2	2
24	poacher pit eff.	300	240	120	4	3*	720	15	TSS	2	2
25	poacher pit eff.	300	240	120	4	3*	720	20	TSS	3	3
26	poacher pit eff.	300	240	120	4	3*	720	30	TSS	4	4
27	poacher pit eff.	300	240	120	4	3*	720	60	TSS	3	3
Phase III - Extended operation											
28	optimum	?	1 month	?	?	optimum	?	optimum	TSS	?	?

Table B2. Experimental data for crossflow microfiltration of NC fines.

Test No.	Hour Meter	Backwash timer (min)	Pressure (psi) PG-1	PG-2	PG-3	PG-4	TMP	Cross flow (gpm)	Flux flow (gpm)	Notes
1	10:19	10	25	22	19	25	1.5	32	0	
	10:27	10	24	22	18	25	1.5	32	2.5	
	10:37	10	24	21	18	25	1	32	2.4	
	10:40	10								Backwash
	10:42	10	24	21	18	25	1	32	2.5	
	10:54	10	25	21	19	25	1	31.5	2.25	
	10:60	10								Backwash
	10:61	10	24	21	18	24	1.5	31.5	2.4	
2	10:65	10	24	22	17	23	3	31	4.9	
	10:73	10	24	22	17	23	3	31	4.75	
	10:75	10								Backwash
	10:77	10	23	20	16	22	2.5	31	4.75	
	10:89	10	25	23	18	25	2.5	32	0	Valve closed off – filtrate recirculated
	10:92	10	24	22	16	27	1.5			
	10:93	10								Backwash
	10:15	10	24	20	16	22	3	31	4.75	
	11:08	10	24	20	16	23	2.5	31	4.4	
3	11:12	10								Backwash
	11:15	10	22	19	12	17	6	29.5	10	
	11:26	10	23	19	12	17	6.5	29	10.3	
	11:31	10	22	20	12	18	6	29.5	10	
	11:42	10	22	20	13	17.5	5.75	29	10	
4	16:97	15	23	23	22	22.5	0.75	30.5	5	
	17:13	15	25	23	22	22.5	1.75	30	5	
	17:21	15	24	22	22	22	1	30.5	5	
	17:38	15	24	22	22	22	1	30.5	5	
	17:46	15	24	22	22	22	1	30.5	5	

Table B2. (Cont'd).

Test No.	Hour Meter	Backwash timer (min)	Pressure (psi) PG-1	PG-2	PG-3	PG-4	TMP	Cross flow (gpm)	Flux flow (gpm)	Notes
16	54.83	60	24	22	21	22	1.5	30.5	4	
	55.06	60	25	22.5	21.5	22.5	1.75	30.5	4	
	55.27	60	25	22.5	21.5	22.5	1.75	30.5	4	
	55.51	60	25.5	22.5	21	22.5	2.25	30.5	4	
	55.63	60	25.5	22.5	21	22.5	2.25	30.5	4	Flux slightly less
	55.67									Backwash – reset timer
	55.69	60	24.5	22.5	21.5	22.5	1.5	30.5	4	
	56.03	60	25	22.5	21	22.5	2	30.5	4	
	56.22	60	25	22.5	21	22	2.25	30.5	4	Flux slightly less
	56.39	60	25	22.5	21	22	2.25	30.5	4	Flux slightly less
	56.52	60	25.5	22.5	21	22	2.5	30.5	4	Flux slightly less
	56:61	60	25	22.5	21.5	22	2	30.5	4	
	56:84	60	25	22.5	21	22	2.25	30.5	4	
	57:00	60	25	22.5	21	22	2.25	30.5	4	Flux slightly less
	57:18	60	25	22.5	21	22	2.25	30.5	4	Flux slightly less
	57:39	60	25	22.5	21	21.5	2.5	30.5	4	Flux slightly less
	57:44									Backwash
	57:45	60	24.5	22	21	21.5	2	30.5	4	
	57:79	60	25	22	21	22	2	30.5	4	
	58:01	60	25	22.5	21	22	2.25	30.5	4	Flux slightly less
	58:16	60	25	22	21	22	2	30.5	4	Flux slightly less
	58:26	60	25	22.5	21	21.5	2.5	30.5	4	Flux slightly less

Appendix C: Budget Proposal for Removal of NC Fines From the Delaval Centrifuge Effluent by Crossflow Microfiltration

MEMCOR

[illegible] Greenspring Drive
[illegible] MD [illegible]
Telephone (301) 252-[illegible]
Telefax (301) 252-6027

January 24, 1992

Hercules Incorporated
Radford Army Ammunition Plant
Post Office Box 1
Radford, VA 14141-0100

ATTN: M.C. Alderman

REF: RA39184

Dear Sir:

Based on the requirements outlined in the above referenced inquiry, we have prepared a budget proposal for a Memcor® system capable of handling a design flow of 1MGPD.

The proposal identifies the basic components and scope of responsibility regarding the proposed Memcor® system and outlines budget pricing for initial capital outlay and yearly operating cost.

A more detailed proposal with supporting documentation would be forthcoming once project details, technical requirements and project milestones are specifically defined.

We appreciate Hercules' continuing interest in the use of the Memcor® technology at RAAP and look forward to the opportunity to install a full scale system at your facility.

Should you have any questions regarding the attached information, please do not hesitate to contact us.

Sincerely,
MEMCOR

J. R. Schiedel

John R. Schiedel
Sales

JRS/met
Enclosure

Project: Hercules Inc. RA39184 **Date:** January 24, 1992
No.: JS92-01003

BUDGET PROPOSAL FOR RADFORD ARMY AMMUNITION PLANT

Project Description

Provide a microfiltration plant with a 1MGPD capacity capable of separating and recovering nitrocellulose fines from existing process. Filtrate from the system will also be of sufficient quality to be considered for reuse and/or discharge and will exceed water standards outlined by the new regulations mandated by the U.S. Environmental Protection Agency (USEPA) for turbidity, levels of suspended solids, etc.

Basic Approach/Equipment Recommendation

Memcor® is recommending its high flow Continuous Microfiltration (CMF) system comprised of ten square meter membrane bundles arranged in compact modular arrays. Systems are designed with a unique automated gas backwash regime allowing for continuous high flow rates and long on stream service life. Design assures optimal performance with on-line system integrity capability. Proposed system utilizes a crossflow mode of operation to prevent membrane fouling and assures high flux rates.

The proposed system has been sized to permit for sufficient capacity to provide both redundancy and allowances for backwash and CIP modes of operation. Basic approach permits for two systems on line/ one in standby configuration.

A total of three (3) six hundred square meter systems is recommended. Each $600M^2$ block (Type 600M10) consists of sixty $10M^2$ modules. Pump type Goulds 3171 or equal (submersible) will supply driving force to operate systems in both forward and backwash modes of operation. Proposed microfiltration plant will include, in addition to basic membrane/module block, appropriate manifolds for system connections, pumps, air compressors, software, process/control design criteria, overall design strategy, backwash/ CIP tanks, etc. Control panels and electrical components will meet NEMA 4 standards. Also included in budget price are provisions for commissioning, training, warranty and detailed drawings pertaining to orientation of process equipment.

Note: It is recognized that a properly sized automated prefilter device will lead to optimization of the Memcor technology. It is suggested that an automated strainer such as that offered by Ronnigon Petter or Hayward Strainer be considered. This particular requirement is outside of Memcor®'s scope of supply.

Project: Hercules Inc. RA39184 **Date:** January 24, 1992
No.: JS92-01003

Scope of Supply by Memcor®

A. Based on the design flow of 1MGPD noted for the RAAP facility, Memcor® proposes the following microfiltration plant package.

- Design Flow : 1 MGPD
- Recommended Approach : Microfiltration plant, Type 600M10-3/Modular

	Major Components	Quantity	Description
1.	Microfiltration blocks	Three (3) 600M10	Each consisting of sixty (60) ten square meter modules mounted on a frame with appropriate manifolds, etc. (See attached sketches on the recommended system).
2.	Backwash Tank	1	2500 gallon tank design to accept backwash for on-line operating system.
3.	Clean in place CIP system	1	Automated system which dispenses chemcials when system requires off line cleaning.
4.	Air Compressors	3	Required to supply initial air source necessary for standard gas backwash. Two on line; one standby.
5.	Air Receivers	2	Part of air supply system for backwash regime.

Project: Hercules Inc. RA39184 **Date:** January 24, 1992
No.: JS92-01003

Major Components	Quantity	Description
6. Pump	4	Submersible pumps (Goulds type or equal) required to feed Memcor system. Attached schematic (conceptual) drawing assumes placement of pump in external sump, pond, etc.
7. Controls		Integration of components described above.
8. Design Strategy		Includes process control strategy and overall layout recommendations.

. Budget Price: $1,375,000.00

B. Estimated Yearly O & M Cost

Major Components

Modules	- $45,000/year
Cleaning Chemicals	- $25,000 (Based on once/month cleaning)
Electrical	- $65,000
Maintenance	- minimal
Labor	- minimal

Note: (1) While the system is automated, some labor cost associated with routine maintenance, equipment monitoring, and cleaning will be incurred. Estimated cleaning cycle would be once a month.

(2) Yearly membrane module costs are based on a 3 year membrane life. (Replacement consists of removing fiber bundle from module assembly rather than entire system).

(3) Above O & M costs are estimates.

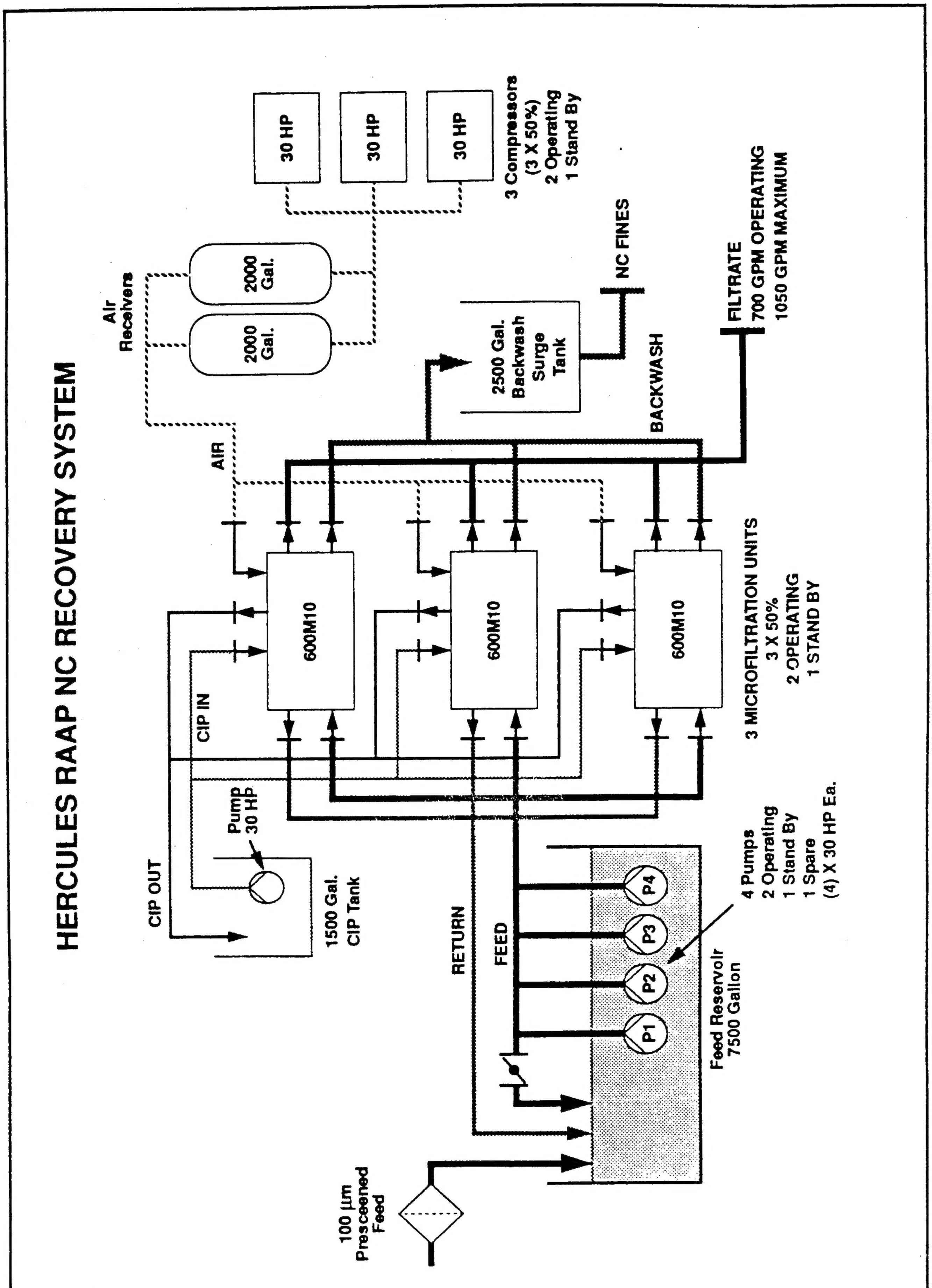
Project: Hercules Inc. RA39184 **Date:** January 24, 1992
No.: JS92-01003

Exclusions:

Above budget prices do not include general civil, electrical, mechanical or overall project arrangement cost. Contingencies such as rework, penalties and miscellaneous expenses are also not included.

A layout sketch along with an engineering spec sheet on the 600M10 is attached for your information. This is a bacis design concept only.

Hopefully, the above information will assist you with your planning on the installation of a Memcor® package microfiltration plant. Should you require further data please let me know.


HERCULES RAAP NC RECOVERY SYSTEM
30 HP
30 HP
30 HP
3 Compressors
(3 X 50%)
2 Operating
1 Stand By
Air Receivers
2000 Gal.
2000 Gal.
AIR
NC FINES
2500 Gal. Backwash Surge Tank
BACKWASH
FILTRATE
700 GPM OPERATING
1050 GPM MAXIMUM
CIP IN
CIP OUT
Pump 30 HP
1500 Gal. CIP Tank
600M10
600M10
600M10
3 MICROFILTRATION UNITS
3 X 50%
2 OPERATING
1 STAND BY
RETURN
FEED
P1
P2
P3
P4
4 Pumps
2 Operating
1 Stand By
1 Spare
(4) X 30 HP Ea.
Feed Reservoir
7500 Gallon
100 µm
Prescreened
Feed

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ATTN: Facilities Engr (3) 85613

USA Engr Activity, Capital Area
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ATTN: SMCAR-ISE

Engr Societies Library
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Defense Nuclear Agency
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Defense Logistics Agency
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Walter Reed Army Medical Ctr 20307

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416th Engineer Command 60623
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